august 1958

nlgi spokesman

journal of the national lubricating grease institute

Sensory Examination of Lubricating Greases

By ALBERT S. ORR

The Mixing Efficiency of Grease Kettles

By A. BEERBOWER, E. O. FORSTER, J. J. KOLFENBACH and H. G. VESTERDAL

Pyrogenic Silica-Thickened Greases

By PETER B. MARSDEN

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Future Meetings

AUGUST, 1958

10-15 National Congress of Petroleum Retailers, Inc., 12th Annual Session, McAllister Hotel, Miami, Florida.

11-14 SAE, national west coast meeting, Ambassador Hotel, Los Angeles.

SEPTEMBER, 1958

8 NLGI Board of Directors meeting, New York City, location to be announced.

10-12 National Petroleum Association, 56th Annual Convention, Traymore Hotel, Atlantic City, N. J.

21-24 AIME, Conference on Petroleum, Cosmopolitan Hotel, Denver, Colo.

24-25 Oil Petroleum Marketers Association Fall Conference and Golf Tournament, Dayton Biltmore Hotel and Walnut Grove Country Club, Dayton, Ohio.

28-30 IOCA Eleventh Annual Meeting, Palmer House Hotel, Chicago, Ill.

OCTOBER, 1958

6-7 Packaging Institute, Petroleum Packaging Committee, Sheraton - Fontenelle Hotel, Omaha.

12-14 Oil Progress Week

13-15 ASLE-ASME Joint Lubrication Conference, Hotel Statler, Los Angeles, Calif.

20-22 SAE National Transportation Meeting, Lord Baltimore Hotel, Baltimore, Md.

22-24 SAE National Diesel Engine Meeting, Lord Baltimore Hotel, Baltimore, Md.

27-29 NLGI Annual Meeting, Edgewater Beach Hotel, Chicago, Ill.

NOVEMBER, 1958

5-6 SAE, national fuels and lubricants meeting, the Mayo Hotel, Tulsa.

6-8 National Oil Jobbers Council, Conrad Hilton Hotel, Chicago.

10-12 API annual meeting, Conrad Hilton Hotel, Chicago.

30-Dec. 5 ASME, annual meeting, Statler and Sheraton-McAlpine hotels, New York City.

JANUARY, 1959

25-27 ASLE gear symposium, Hotel Morrison, Chicago.

FEBRUARY, 1959

2-6 ASTM National Meeting, William Penn Hotel, Pittsburgh, Pa.

***MARCH, 1959**

3-5 SAE Passenger Car, Body, and Materials Meeting, Sheraton-Cadillac, Detroit, Mich.

APRIL, 1959

21-23 ASLE Annual Meeting and Exhibit, Hotel Statler, Buffalo, New York.

MAY, 1959

15-24 International Petroleum Exposition

31-June 6 Fifth World Petroleum Congress.

JUNE, 1959

14-19 SAE Summer Meeting, Chalfonte-Haddon Hall, Atlantic City, N. J.

*Tentative.

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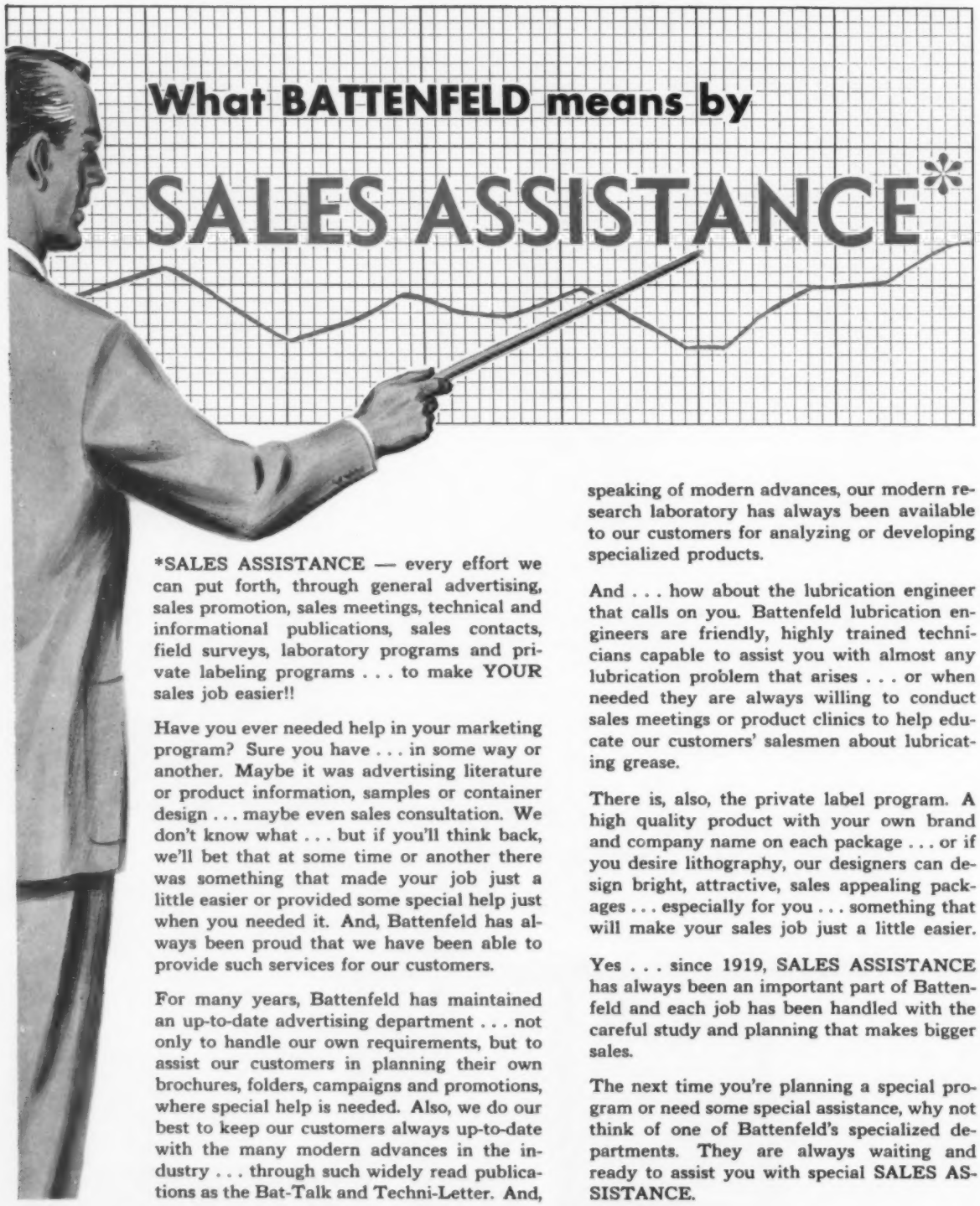

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*SALES ASSISTANCE — every effort we can put forth, through general advertising, sales promotion, sales meetings, technical and informational publications, sales contacts, field surveys, laboratory programs and private labeling programs . . . to make YOUR sales job easier!!

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For many years, Battenfeld has maintained an up-to-date advertising department . . . not only to handle our own requirements, but to assist our customers in planning their own brochures, folders, campaigns and promotions, where special help is needed. Also, we do our best to keep our customers always up-to-date with the many modern advances in the industry . . . through such widely read publications as the Bat-Talk and Techni-Letter. And, speaking of modern advances, our modern research laboratory has always been available to our customers for analyzing or developing specialized products.

And . . . how about the lubrication engineer that calls on you. Battenfeld lubrication engineers are friendly, highly trained technicians capable to assist you with almost any lubrication problem that arises . . . or when needed they are always willing to conduct sales meetings or product clinics to help educate our customers' salesmen about lubricating grease.

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NLGI PRESIDENT'S PAGE

By R. CUBICCIOTTI, *President*

Salesmen and Scientists Complement Each Other

I have alluded on separate occasions in the past to the importance of stepping up our selling efforts and to the pride the members of our industry can take in the results of their research efforts.

The allusions may seem to be disparate; actually, they are closely interrelated. Members of the Institute would do well to devote more than passing attention to ways of wedding the art of selling to the science of research in the best interests of the lubricating grease industry and the public.

For what better way is there to capitalize on the advances that our researchers and our scientists have made in the development of grease lubricating products? What better way to give the consumer a clearer understanding of what is being done by us for his benefit?

It is perfectly obvious, of course, that even the best efforts of the best research scientists may have very little chance of seeing the light of day unless they are properly "sold" to the consumer. But it is not so obvious how to determine the exactness with which we should define and channel the roles and interrelationships of scientist and salesman.

The answer, perhaps, is in establishing an awareness on the part of both the research staff and the sales force on the role that each has to play. The first must have a knowledge of what our customers want and need; the second, a realization of what the scientific advances in our products mean to the customer.

Progress means change and change means problems—to the consumer, to the industry and to the people in it.

The researcher, who is equipped to solve these problems in so far as they relate to industry, can better appreciate what they mean to the consumer if he has a proper working relationship with the salesman. The latter is better able to serve his customer if he gets from the researcher an understanding of what the changes involve.

In other words, scientist and salesman complement each other.

Only by carefully defining and integrating the roles of the two and by giving both a broad picture of the potentialities of the entire industry, will we be able to reap the full benefit of their joint efforts.

We are living in an age of science; we must make a science of selling in order to convey with maximum effectiveness to the consumer the importance of the lubricating grease industry in this scientific age. ■

NOW TRY STURACO XL

...Stuart's new and improved SULPHUR-CHLORINE-LEAD BASE for automotive gear lubricants

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Stuart's Sturaco XL Base is a new and improved sulphur-chlorine-lead base for use in the compounding of heavy-duty gear lubricants meeting today's modern automotive service requirements.

Sturaco XL Base has been developed as a result of Stuart's continuous research program on automotive gear lubricants. We have been a leading supplier of finished gear lubricants to Ford Motor Company (M2C28A & B; M4642D & E), General Motors Corporation (GM4735M), and the Chrysler Corporation (MS-1220)—your assurance of uniform, high quality from Sturaco XL Base.

For complete technical data and prices, write today.

Sturaco XL Base specifications

Sulphur	4.8% Min.
Chlorine	4.2% Min.
Lead	6.0% Min.
Saponifiable	40% Min.
API Gravity	10-12
Pounds per Gallon	8.22 to 8.33

Timken Test

10.0% by weight of Sturaco XL Base in SAE 90 stock carries 50 pound minimum Timken machine lever load.

28.5% by weight of Sturaco XL Base in SAE 90 stock carries 80 pound minimum Timken machine lever load.

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AUTOMOTIVE LUBRICANTS DIVISION

nlgi spokesman

Volume XXII AUGUST, 1958 Number 5

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THE COVER

ILLUMINATED inspection plates for lubricating greases are one way to catch foreign matter in the product before shipment . . . this is just one method of sensory examination as listed by author Albert S. Orr, in his article beginning on page 221. Because so many customers judge a lubricating grease product by sight, smell and touch, Mr. Orr has proposed a standard vocabulary of terms for describing the sensory observations, the first time such a proposal has been made through the pages of the NLGI SPOKESMAN. We invite your attention to this piece.

The NLGI SPOKESMAN is indexed by Industrial Arts Index and Chemical Abstracts. Microfilm copies are available through University Microfilm, Ann Arbor, Mich. The NLGI assumes no responsibility for the statements and opinions advanced by contributors to its publications. Views expressed in the editorials are those of the editors and do not necessarily represent the official position of the NLGI. Copyright 1958. National Lubricating Grease Institute.

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More and more, industry depends on the clear, moisture-free lubricating greases based on Metasap Metallic Soaps. Chances are you already use Metasap Metallic Soaps in your products. If not, don't delay any longer. Write today for complete details. And remember, behind all Metasap products stands Metasap Technical Service—ready to attend promptly to all inquiries and requests. Metasap Chemical Company, Harrison, N.J.

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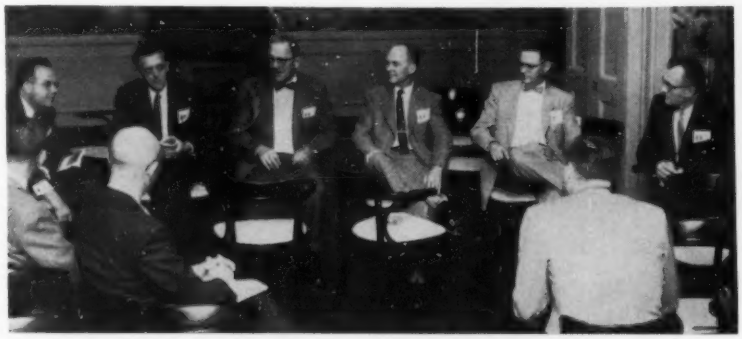
News About NLGI

1958 Annual Meeting Will Offer Unique Session for Technical Men—a Rheology Symposium



FIRST plans for Fundamental Research program were made at Chicago last year.

Technical men attending the 26th Annual Meeting of the National Lubricating Grease Institute in Chicago next October will benefit from a symposium on rheology of lubricating greases which will be the most comprehensive symposium on this subject ever presented by any organization, to date.

The two-and-a-half day meeting at the Edgewater Beach hotel is planned for Monday, Tuesday and Wednesday, October 27, 28, and 29, and the rheology symposium is one highlight in a series of well planned sessions for the industry, by F. E. Rosenstiehl, program chairman, and his committee.

General theme of the meeting is "Lubricating Grease in Industry" and for the first time, a joint session will be held with a parallel group—the American Gear Manufacturer's association, during one afternoon section of NLGI's meeting. Some of the tentative titles of talks during the annual meeting:

industrial gear design . . . industrial gear lubrication probblems from gear manufacturers, dispensing equipment manufacturers, lubricant manufacturers and the consumer standpoints . . . recent developments in industrial gear lubricants...a new preformed thickener for grease . . . facts and factors in grease manufacturing costs . . . function of technical service in grease development and application . . . . gas chromatography in analysis of fatty acids . . . milling and dispersion of grease, and several others.

Because the rheology symposium is considered "pure" research, this will be offered at a separate meeting, which will run concurrently with the general meeting. The symposium is sponsored by the Institute's newest Technical sub-committee, the Fundamental Research group under the chairmanship of J. J. Kolfenbach.

In addition to the NLGI-AGMA joint meeting and the symposium, the regular Technical Committee meeting will be held as usual on Wednesday morning, October 29, under the guidance of T. G. Roehner, chairman. The traditional social hour and banquet functions will be offered on Tuesday evening, October 28.

New Representatives Announced by Member Firms

Girdler Process Equipment division of the Chemetron corporation (formerly National Cylinder Gas) has named Mr. H. E. Huber, sales manager of process plants as their NLGI Technical Representative.

Continued, page 219

Continental puts extra service into every steel container

From advanced research to faster delivery, Continental container service smooths your shipping problems

Continental gives you the highest quality steel containers *plus* the benefits of famous Continental service. You deal with a Continental expert who knows the problems of the petroleum industry. You order from a complete line of steel containers, superbly lithographed for powerful sales appeal. You get fast delivery of all the containers you need, when you need them. And if you have a special problem, Continental's research and engineering services help you solve it. Let us show you what we mean by famous Continental service. Call today.

Exclusive Perma-Lining enamels provide 100% protection for hard-to-hold petroleum and chemical products. Perma-Lined containers are airless hot sprayed *after fabrication* to prevent corrosion-causing cracks and assure 100% complete interior coverage. In tight or open head pails—with lug, ring seal or lever lock covers.

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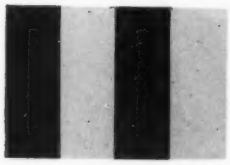

News About NLGI—cont'd

Continued from page 217

Oil-Kraft, Inc. has appointed Mr. Robert L. Peters, president, as the firm's NLGI Technical Representative.

British Petroleum Company, Ltd., named Mr. Leslie S. Thornes to fill that company's position as NLGI Company Representative.

Enjay Company, Inc. has appointed Mr. Brian Casey, product manager for additives, as their Company and Technical Representative to NLGI.

Metasap Chemical corporation named Mr. T. J. Campbell, sales manager, as NLGI Company Representative.

The Pennzoil company has made Mr. Elmer Lemire, service engineer their new NLGI Technical Representative.

Bryant Chemical Co. Changes Name

The name of the Bryant Chemical company has been changed to Cadillac Oil company . . . the address remains the same . . . 13650 Helen Avenue in Detroit. Bryant Chemical is now a subsidiary sales department of Cadillac. Mr. C. P. Bryant is the president of Cadillac Oil, and the NLGI Company Representative.

New Manual in Progress

Work by NLGI committee is progressing on a new manual entitled *Recommended Practices for the Lubrication of Passenger Car-Type Ball Joint Front-End Suspensions.*

Like its predecessor before it, the widely distributed *Recommended Practices for Lubricating Front Wheel Bearings*, the Ball Joint manual is the product of many hands and is now in the process of being checked and rechecked by a joint committee of both technical and marketing men of NLGI.

Because the manual may well get sales over the world like the Wheel Bearing booklet did (currently, over 110,000 copies sold), extreme care is being taken in the preparation. Further developments will be covered in later issues of the NLGI SPOKESMAN.

NLGI Board Members to Meet September 8

There will be a meeting of the Board of Directors of the National Lubricating Grease Institute on Monday, September 8, in New York City. The governing body of the Institute will consider final plans for the 26th Annual Meeting, as well as several other items of business.

SERVICE AIDS

Send Orders to: National Lubricating Grease Institute, 4638 Nichols Parkway, Kansas City, Mo.

REPRINTS — From the NLGI SPOKESMAN are available at low cost. Page forms are left standing for three months, company imprint or advertising arranged.

WHEEL BEARING MANUAL — "Recommended Practices for Lubricating Automotive Front Wheel Bearings." More than 90,000 copies of this booklet have been distributed throughout the world. Fifteen cents a copy (NLGI member price) with quantity discounts — company imprint arranged.

VOLUME XX — Bound volume of the NLGI SPOKESMAN from April, 1956 through March, 1957. An excellent reference source, sturdily bound in a handsome green cover . . . $7.00 (NLGI member price) and $10.00 (non-member) plus postage.

VOLUME XXI—Bound volume of the NLGI SPOKESMAN from April, 1957 through March, 1958. Contains 34 articles and features dealing with lubricating greases and gear lubricants . . . $7.00 (NLGI member price) and $10.00 (non-member) plus postage.

NLGI MOVIE — "Grease, the Magic Film," a 16-mm sound movie in color running about 25 minutes, now released. First print $600, second print $400, third and subsequent orders $200 each (non members add $100 to each price bracket).

BONER'S BOOK—Manufacture and Application of Lubricating Greases, by C. J. Boner. This giant, 982-page book with 23 chapters dealing with every phase of lubricating greases is a must for everyone who uses, manufactures or sells grease lubricants. A great deal of practical value. $18.50, prepaid.

Available!

Manufacture and Application of LUBRICATING GREASES

by C. J. Boner

Chief Research Chemist
Battenfeld Grease and Oil Corp.

1954
982 pages
$18.50 prepaid

982
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IN THESE
23
BIG CHAPTERS

1 Introduction
2 Structures and Theory
3 Additives Other Than Structural Modifiers
4 Raw Materials
5 Manufacturing Processes
6 Equipment for Lubricating Grease Manufacture
7 Aluminum Base Lubricating Greases
8 Barium Base Lubricating Greases
9 Calcium Base Lubricating Greases
10 Lithium Base Lubricating Greases
11 Sodium Base Lubricating Greases
12 Lead Soap Lubricating Greases
13 Strontium Base Lubricating Greases
14 Miscellaneous Metal Soaps as Components of Lubricating Greases
15 Mixed Base Lubricating Greases
16 Complex Soap Lubricating Greases
17 Non-Soap Thickeners for Lubricating Fluids
18 Fillers in Lubricating Greases and Solid Lubricants
19 Residua and Petrolatums as Lubricants
20 Analysis of Lubricating Greases
21 Tests of Lubricating Greases and Their Significance
22 Application of Lubricating Greases
23 Trends in Lubricating Greases

Here in one giant volume . . . the most complete storehouse of information ever published on the composition, properties and uses of lubricating greases!

The book begins by describing in detail the structure and theory of lubricating greases. Then follow chapters on the various raw materials, processes and manufacturing equipment. Lubricants containing specific thickeners, including such recent developments as lithium soaps, complex soaps and non-soap gelling agents, receive special attention.

Of major interest is the large section on present uses and future trends of lubricating grease products. Here you'll find the complete details of when, where, and how to apply a specific lubricant for any given purpose.

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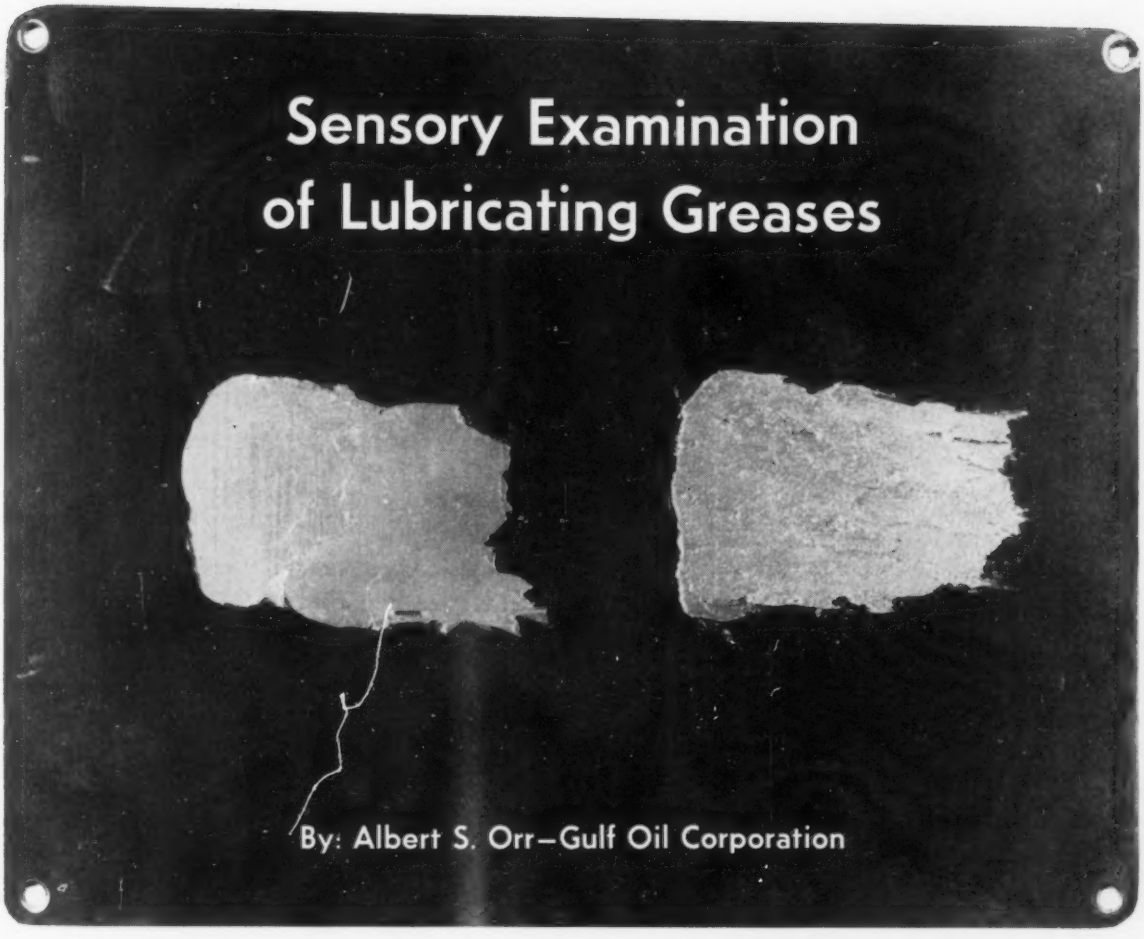
Sensory Examination of Lubricating Greases

By: Albert S. Orr—Gulf Oil Corporation



Polished steel plate—sample with soap particles at right.

CUSTOMERS OFTEN JUDGE lubricating greases, at least in a preliminary way, by what they can see, feel, smell and possibly taste when a package is opened. This first impression of the grease, like first impressions of people, is often difficult to dispel and may adversely influence the customers' opinion despite excellent performance in actual service.

When the cover is removed from a pail or drum of grease, the customer looks at the surface of the grease and forms a general opinion as to the color and overall appearance of the grease. He will note any free oil on the surface of the grease or in depressions. He may use a spatula or putty knife to spread a thin film of grease on some smooth surface and observe whether it contains soap particles or visible foreign matter. If a grease is not similar in color and general appearance to earlier receipts of the same product, it is immediately suspect and may be refused.

Almost universally the grease user will place a small portion of the grease between the thumb and forefinger and pull it apart and push it together a few times. From this he can note the texture of the grease and may form an opinion as to whether it is too hard or too soft for his use. The thumb and forefinger test will show the texture of a lubricating grease as well as any formal laboratory test now available, but this test should not be used as a substitute for the penetration test for determining the consistency of a grease.

The grease user cannot avoid smelling a grease when he opens a pail or drum and will, of course, complain of any unusual or objectionable odor. Perfumes or maskants were once widely used to cover up unpleasant odors in greases, but this practice has almost disappeared.

In the past, many grease makers and some customers would taste a grease to determine whether it was alkaline or acidic. This is not recommended as more precise laboratory methods are available for determining

acidity, and some of the components of modern greases may be harmful when placed in the mouth. Although lubricating greases do not usually contain highly toxic materials, they are not to be eaten.

The results of sensory examinations of greases have at times been described in rather picturesque terms. These terms may vary widely among different persons, thus leading to misunderstandings.

The purpose of this paper is to propose a standard vocabulary of terms for describing the sensory observations of a lubricating grease. Some of the methods and equipment used by one grease maker to assure shipment of greases having uniformity of color, appearance, texture and odor are presented as the background for this discussion.

Color

There is no readily available or generally accepted method for determining colors of lubricating greases and expressing colors by numerical values as is done with lubricating oils. Thus, the grease manufacturer and user must use words in describing the color of the product. Lubricating greases can be made in almost any shade and intensity of color through use of dyes, fillers, non-soap thickeners and other additives. However, the color of most conventional soap-thickened greases is controlled by the color of the lubricating oil base and the metallic soap. Such greases may be opaque or clear, depending on the type of soap and other variables in composition and processing. The color of opaque greases may best be described as some shade of brown. For clear grease, some shade of amber seems more suitable. It is suggested that the shade or intensity be characterized as very light, light, medium, dark and very dark. The very light shade would represent only a faint brown for an opaque grease and a faint amber (yellow) for a clear grease. The very dark shade would apply to a grease that is almost black.

In many greases, the bloom or fluorescence of the mineral oil component is visible and may affect the apparent color. If so, this should be noted in describing the color. For instance, a grease containing Pennsylvania cylinder stock may have a pronounced green bloom. The color of such a grease might be described as dark brown—green bloom. On the other hand, a grease made from a naphthenic oil might have a discernible blue bloom, and its color could be described as light amber—blue bloom. If no bloom is readily apparent, no mention is made of bloom in describing the color.

It is important that all greases be examined for color and bloom by reflected light, preferably daylight, and in metal containers—sample cans, pails or drums. The color of greases in glass sample jars may, at times, be misleading, particularly when observed through the glass. This may be due to reflection from the glass or to transmission of light through the grease. In most cases, the customer observes the color of grease in a metal container.

Greases containing fillers, dyes and some of the new non-soap thickeners may have colors matching almost any hue of the rainbow. At extremes a grease containing a substantial amount of graphite is black, while a grease containing zinc oxide may be almost white. Dyes of almost any color may be used. Sometimes this is done to give the grease a distinctive color for ready identification. On the other hand, some of the newer thickeners are actually colored pigments or dyes which will impart a characteristic color to the grease. In many cases where fillers, dyes and colored thickeners are used, the color of the oil base will modify to some extent the color imparted by the filler, dye or thickener. When grease colors cannot adequately be characterized in terms of brown or amber as suggested above, the color should be described in terms of the predominant color with adjectives to indicate shade and intensity, and also, any less predominant color that is visible. For example, a grease dyed dark red may have

FIGURE 1, texture—buttery

FIGURE 2, texture—short fiber

FIGURE 3, texture—long fiber

FIGURE 4, texture—stringy

a brownish tinge due to the color of the oil base and soap, and should thus be described as a dark brownish red grease.

The grease maker's main problem with regard to color is to assure that all shipments of a given grease have a uniform color. One grease maker handles this by preparing a set of color standards by selecting samples from the lightest and darkest colored batches of each grade that are considered acceptable. These samples are put up in small cans and retained in the laboratory for comparison with each production lot of that particular grease. A sample of the production lot is placed in an identical sample can and compared with the light and dark standards for this particular grease in daylight or under a daylight lamp. To be acceptable for shipment, the production lot must be no lighter in color than the light standard and no darker than the dark standard. Usually, the color of a production lot will fall between that of the two standards. Since some greases may change color on aging, the standard samples are replaced at about yearly intervals by again selecting typical production lots for standards.

Appearance

When a customer removes the cover from a package of grease and looks at it, he may make a number of other observations in addition to the color of the grease. He probably will note whether the surface of the grease is smooth or rough, whether there are cracks in the surface and whether separated oil is present. He may also note whether the grease has a bright or dull luster. If a thin film of the grease is spread on a smooth surface using a spatula or other suitable tool, he may observe whether the grease is grainy or smooth and whether it contains visible soap lumps or foreign matter. It is proposed that the word "appearance" with regard to grease include all of the visual observations that might be made on examining the surface of a grease in the final package and on examining a thin film spread on a smooth surface. Thus, it would include all of the items shown in Table I.

One grease maker uses a somewhat more elaborate thin film examination than is normally employed by customers. The main purpose of this is to detect soap particles and foreign matter that might appear in the finished grease due to failure of the screen in the filling line. An illuminated inspection plate as shown on cover illustration is used. This consists of a triangular piece of opal glass illuminated from below by a single daylight fluorescent tube which is enclosed in a box on which the glass plate rests. A sample of the grease to be examined is spread on the glass plate with a spatula to give a thin uniform film. With the light on, soap particles and foreign matter can be readily detected, and if found, will be cause for rejection of a batch of grease. Generally, at least three samples are taken during filling out of a kettle of grease. These will be from the first, middle and last packages, and all must be free from visible soap and foreign matter and uniform with respect to color and graininess for the entire batch to be shipped.

For certain greases, particularly semi-fiuid oils, transparent soap lumps are more readily detected if the grease is examined by reflected light. In these cases, the sample is spread in a thin film on a polished stainless steel plate for examination under a daylight lamp. A given grease may be examined both by transmitted light as described above and by reflected light on the steel plate. Discernible soap particles or foreign matter by either procedure is cause for rejection or further processing.

These tests are not used as a substitute for the ASTM method of Test for Deleterious Particles in Lubricating Grease, ASTM D 1404-56T, which will detect some particles not readily visible and allows a semi-quantitative estimate of the number of particles which might prove at least slightly abrasive in service.

Texture

When a customer examines a container of grease, he will almost subconsciously place a small portion of the grease between his thumb and forefinger and press together and pull apart a few times. From this simple procedure, the customer gets an idea of the texture of the grease. The following terms are recommended for describing texture:

Buttery—separates in short peaks with no discernible fibers.

Short Fiber—short "break- off" with evidence of fibers when portions are separated.

Long Fiber—has a tendency to stretch out into a long single fiber when portions are separated.

Stringy—has a tendency to stretch or string out with no evidence of fiber structure when portions are separated.

Resilient—capable of withstanding compression between thumb and forefinger without permanent deformation or rupture.

Brittle—has a tendency to rupture or crumble when compressed between thumb and forefinger.

Many other terms have been used to describe grease texture. Among these are smooth, grainy, feathery, ropey, crumbly, rubbery, tacky, etc. As noted above, we would like to reserve "smooth" and "grainy" for use in describing the visual appearance of a grease when examined in a thin film. It is believed that the six terms proposed above will satisfactorily describe the texture of any grease that may be encountered and for the sake of uniformity, we would suggest their exclusive use instead of variations such as feathery, ropey, crumbly, rubbery, tacky, etc.

Odor

Lubricating greases may vary widely in intensity and type of the odor that is encountered on opening a package. Most freshly prepared, high quality, conventional soap thickened greases will have little or no odor. Some greases tend to become rancid on aging while others contain additives or other ingredients that impart typical odors. Some odors may be pleasant while others may be disagreeable, though this is to some extent, a matter of individual preference and depends partly on the intensity of the odor and partly on the type.

In describing the odor of a grease, an attempt should be made to indicate both its intensity and its type. The following intensity scale is suggested:

None—no odor detectable.

Slight—odor would not be detected by the average customer but would be detected by an experienced observer.

Mild—odor might be detected by average customer but only if his attention were called to it.

Noticeable—odor readily detected by customer.

Strong—odor that would force itself on attention of customer and possibly render the grease unsuitable for use in confined areas.

The types of odor that may be encountered will vary widely and in general are related to the ingredients used in making the grease. Most commonly, there will be the rather mild odor associated with petroleum lubricating oils. This may be more intense with less highly refined oils and lower viscosity oils. A pronounced kerosene or gasoline odor may indicate contamination and should be noted and investigated. The fat from which the soap is made may impart some odor to the grease, though generally, this is mild if high quality fats are used. Some fats and the soaps made from them may become rancid on aging, and develop a characteristic acidic odor. Many of the additives used in greases have characteristic odors. Some anti-oxidants may have a phenolic or medicinal odor. Some extreme pressure additives may have a sulfury odor similar to hydrogen sulfide or sulfur dioxide. Lead naphthenates usually have a characteristic naphthenic acid odor.

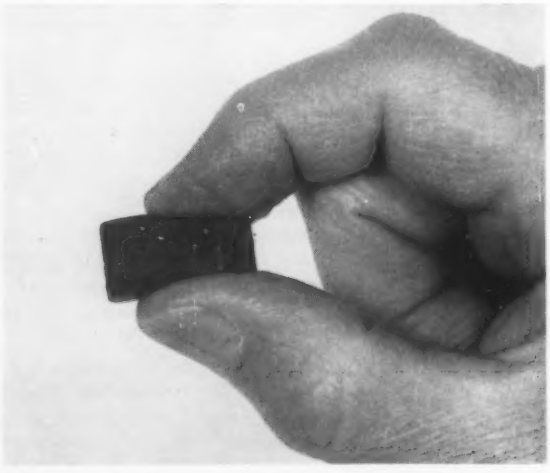


FIGURE 5, texture—resilient

FIGURE 6, texture—brittle

Often, an experienced observer can identify the odor as resulting from, or being similar to, a specific compound such as nitrobenzene, oil of wintergreen or other scenting agent. Some of the greases containing non-soap thickeners have characteristic odors due to the modifying agent used. For instance, some bentone greases have a noticeable odor of acetone or methyl alcohol.

The type of odor should be described in accordance with the following table amplified if possible by reference to a specific chemical compound or product:

Type of Odor	*Examples*
Petroleum	Lubricating oil, kerosene, gasoline
Fatty	Well refined fats, vegetable oils and fatty acids
Flowery	Violet, geranium, and many others
Spicy	Cinnamon, cloves, etc.
Medicinal	Phenol, iodoform, etc.
Rancid	Rancid butter, lower fatty acids
Ammoniacal	Ammonia, alkyl amines
Sulfury	Hydrogen sulfide, mercaptans, sulfurized fats
Burnt	Burnt wood or straw
Acrid	Scorched fat
Grassy	Crushed grass
Musty	Decomposed straw
Decayed	Spoiled meat
Septic	Stale sewage

Some odors of lubricating greases are transitory and disappear or diminish in intensity in a short time after the package is opened. It is suggested that odors be observed immediately after opening the package and then again an hour or two after opening. If there is any difference in type or intensity in the two observations, this should be reported.

Some odors tend to dull the olfactory sense and cannot be detected and identified if too intense. It is recommended that the first sniff of any grease be taken very cautiously immediately after removing the cover from the package by fanning air from the top of the package toward the face.

Of course, in order to properly evaluate the odor of a grease, it is desirable to conduct the examination in an area that is free from other odors.

As noted above, both the intensity and type of odor should be reported using the terms suggested above amplified by reference to specific chemical compounds where these can be identified by the observer.

TABLE I

Proposed Standard Terms for Reporting Visual Examination of Lubricating Greases

APPEARANCE

1. Color
   a. Amber—very light, light, medium, dark, very dark
   b. Brown—very light, light, medium, dark, very dark
   c. Other—specify shade and intensity of predominant color that is visible.
2. Bloom or Fluorescence
   a. Green—note intensity—slight, medium, marked
   b. Blue—note intensity—slight, medium, marked
   c. None
3. Luster
   a. Bright
   b. Dull
4. Surface Examination in Package
   a. Smooth
   b. Slightly rough
   c. Rough
   d. Cracks—note number and size
   e. Separated oil—note approximate quantity
5. Thin Film Examination
   a. Smooth
   b. Slightly rough
   c. Rough
   d. Grainy
   e. Soap particles
   f. Foreign matter

Conclusion

The foregoing discussion outlines the conditions for making sensory observations of lubricating greases as accurate and meaningful as possible. The limitations of such observations are also mentioned.

A vocabulary for describing the results of observations made by sight, touch and smell has been proposed. It is believed that the general use of this vocabulary will eliminate many misunderstandings between customer and grease supplier. ■

About the Author

ALBERT S. ORR, *a native of Texas, obtained his B.S. degree in Chemical engineering from Texas A&M college in* 1924. *Immediately after graduation, he started work for the Gulf Oil corporation in their Port Arthur, Texas laboratory. In* 1930 *he was transferred to Gulf's refinery technology laboratory in Philadelphia, Pa., and became director of that laboratory in* 1950. *In* 1951 *he was transferred to Gulf's main office in Pittsburgh, Pa., and is currently working in the coordinator's group of the manufacturing department handling matters pertaining to product development and product quality. Much of this work has to do with grease development and quality. Mr. Orr is a member of the American Chemical Society, Society of Automotive Engineers, American Society for Testing Materials, and other technical societies.*

Presented at the NLGI 25th annual meeting in Chicago, October, 1957

The Mixing Efficiency of Grease Kettles

By: A. Beerbower—Esso Standard Oil Co.
J. J. Kolfenbach—Esso Research and Engineering Co.
E. O. Forster—Esso Research and Engineering Co.
H. G. Vesterdal—Esso Research and Engineering Co.

THE MANUFACTURE OF greases requires efficient mixing. Past experience has suggested that differences in kettle design, and hence in mixing efficiency, are responsible for the success or failure—as the case may be—of producing satisfactory products. The grease manufacturer has been hampered by lack of adequate means for rating the mixing efficiency of various types of kettles.

Various methods have been used in the past for estimating mixing efficiency. In one of these methods, the rate of temperature rise in the course of manufacture of grease was used as a measure of the mixing efficiency. The results obtained from such heat transfer studies[1] indicated the extent to which grease was removed from the walls of the kettle, and new grease was brought to the walls for heating or cooling, as the case may be. This type of mixing, which is generally referred to as convective mixing,[2] is of great importance to the manufacturer but does not represent the only type of mixing performed in the kettle.

Oil soluble dyes have actually been used to determine the extent of mixing by diffusion and shearing.[2] In this method, numerous samples, taken from different locations in the kettle, had to be analyzed for their dye content, and the concentration of dye found in them was considered to reflect the degree of mixing. The major drawback of such a technique was the large number of samples that had to be analyzed by destructive techniques.

In some instances, the progress of mixing was also followed by measuring the cone penetration of samples taken randomly from the kettle. Results obtained with this method were usually inconclusive because of experimental uncertainties involved in determining penetrations and were of no general value.

With the help of radioactive tracers, it has now become possible to follow closely the progress of mixing without removing large numbers of samples or permanently contaminating the product. In a recently developed method,[3] small amounts of radioactive Iodine 132 are used to follow the progress of mixing. Since the radiation emitted by Iodine 132 penetrates readily through ½ to ¾ inch thick walls, it can be picked up outside the kettle by means of one or several scintillation counters. As mixing progresses, fluctuations in radioactivity decrease and eventually become indistinguishable from inherent fluctuations of the counting device. From this point, the radioactivity detected by the counters decreases according to the decay rate of the Iodine 132, and the time elapsed from the moment Iodine 132 has been added till this point is reached is taken as the mixing time.

Actually, this method determines how long it takes to blend the last ounce of oil uniformly throughout the kettle, and thus affords a rapid means of rating different kettles as to their over-all mixing efficiencies. The differences in mixing time determined by this technique lead to a qualitative understanding of the effect of over-all kettle design on mixing efficiency.

Because of the short half-life of Iodine 132 of 2.3 hours, the grease used in such a study can be safely shipped 24 hours after completion of the test. For the same reason, the health hazards associated with the use of this radioactive material are very small and the safety precautions necessary for working with this material do not interfere seriously with plant operations. Also, Iodine 132 is now readily available and is relatively inexpensive.

Presentation of Data

The mixing efficiency of several types of grease kettles was studied with the aforementioned method developed by Beerbower et al.[3] Radioactive Iodine 132 was added in all cases as an aqueous solution and the progress of mixing followed by scintillation counters mounted outside the kettle, one about 2 feet off the bottom and the other close to the top of the kettle. The scintillation counters were connected to rate meters and recorders on which fluctuations in counting rate could be traced.

A calcium-lithium soap-type multipurpose grease of NLGI Number 2 grade was used throughout this investigation. The base oil used in this product had a viscosity at 210°F. of 70 SUS. The grease was kept at a temperature of 150°F. ± 5°F. throughout the test.

TABLE I

Grease Kettle Characteristics

Kettle Number	1	2	3	*Pilot Plant*
Shape of Kettle Bottom	flat dish	hemispherical	flat dish	flat dish
Ratio of Height/Diameter	0.80	0.90	1.2	0.66 - 1.3
Number of Paddles				
Inner	4	5	3	3
Outer	3	4	3	3
Pitch of Paddles				
Inner	down	up and down	down	down
Outer	up	up	up	up
RPM of Paddles				
Inner	18.5, 37	19.5, 39.3	28	10, 23, 43
Outer	9.0, 18.5	12.3, 24.5	14	10, 23, 43
Charge, Pounds	15,000	15,000	15,000	230 - 350
Volume Swept by Paddles at Top RPM in cu. in. x 10^{-6}/min.	9.4	13.3	5.9	
Ratio of Volume Swept per Min. at Top RPM / Total Volume	17	25	11	16

The study of the mixing efficiency of grease kettles was conducted in two phases. In the first phase, three plant kettles of different design were evaluated and in the second phase the performance of the pilot plant kettle was studied under various operating conditions. The design characteristics of the four kettles are shown in Table I. It will be noted that all are basically of the two-way motion type, but differ in shape and agitation design.

The results of the various evaluations indicate a significant difference in the performances of these kettles, as shown below.

Mixing Efficiency of Grease Kettles

Kettle Number:	1	2	3	*Pilot Plant*
Time for Complete Mixing (Min.)				
At Top Speed	8.0	0.3	42	4.0
At Lowest Speed	11.0	2.75	..	5.8

In analyzing these results, it appears that the following differences in kettle design might significantly affect the over-all mixing efficiency:

1. Kettle shape and height to diameter ratio (H/D).
2. Number and shape of the paddles.
3. The relationship between the speed of the inner counter rotating paddles and the outer frame paddles.

In order to evaluate in more detail the significance of these factors, several runs were made in pilot plant kettle using 2 different H/D ratios (kettle charged with different amounts of the same grease) and three different speeds. The results of these test runs are summarized below.

Pilot Plant Studies

Run Number:	1	2	3	4	5	6
Speed of Paddles, rpm	10	23	43	10	23	43
Charge in Lbs.	——350——			——230——		
Height/Diameter	——1.0——			——.7——		
Time for Complete Mixing, Min.	5.85	5.35	4.0	5.0	3.75	3.12

From these results, it can be gathered that both speed and H/D have an influence on the over-all efficiency in this particular kettle design which is similar to kettles 1 and 3. At the lower H/D ratio, corresponding to a smaller charge of grease, mixing proceeded faster than at the larger H/D ratio. In both cases, increased speed reduced the mixing time.

A mathematical interpretation of these results was attempted and an equation was set up relating mixing time t, in minutes, speed S in rpm, and H/D as follows:

$$\frac{1}{t} = \frac{A}{(H/D)} \cdot (1 - e^{-BS})$$

where A and B are constants. The influence of the H/D ratio on the mixing efficiency has been noticed previously (4, 5) although it entered to the 1.3 power in one case and to the 0.5 power in the other case. The equation developed here can be successfully applied to flat-dish bottom kettles having simple 2-way agitation. A comparison of the calculated and actually measured times is shown in Table II. From this comparison, it can be seen that for the flat-dish bottom

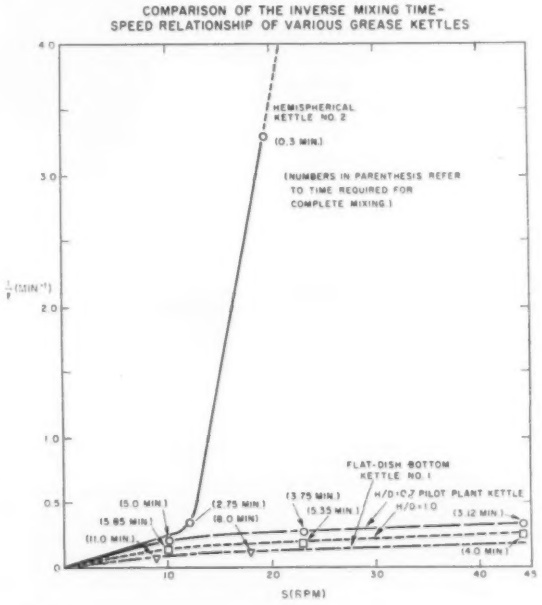


FIGURE 1

kettles, the equation predicts the mixing time in all but one case within ± 15 per cent of the actually observed time. The poor agreement in the case of the hemispherical kettle 2 probably results from the fact this kettle operates on a different mixing pattern. This contention becomes evident if a plot of 1/t versus s (rpm) is compared for the different kettles. This graph is shown in Figure 1. Obviously, the significantly different shape of the time-speed curve of the hemispherical kettle indicates the existence of a different mixing pattern. This difference in mixing pattern is most probably due to the different kettle shape and the fact that the shape of the inner paddles has been changed. While the inner paddles are straight in the other two kettles, they are twisted in kettle 2 so that half of the paddle provides an upward thrust and half of the paddle downward thrust creating a more turbulent mixing. More studies will have to be done to explain more quantitatively the contribution of kettle

TABLE II

Comparison of Calculated and Observed Mixing Times

Kettle	*H/D*	*Speed (RPM.)*	*A*	*B*	*Mixing Time (Minutes)* *Calculated**	*Observed*
Pilot Plant	0.7	10	0.27	0.06	5.75	5
		23			3.42	3.75
		43			2.8	3.12
	1.0	10	0.12	0.1	8.25	5.8
		23			4.9	5.3
		43			3.98	4.0
No. 1	0.8	9.25			11.2	11.0
		18.50			8.08	8.0
No. 2	0.9	12.3	7.2	0.003	3.4	2.75
		19.5			2.5	0.3
No. 3		No calculations were made because of insufficient data.				

*Using Equation Shown in Text.

shape and paddle design to the improved mixing efficiency of the kettle 2.

Conclusions

The results of this study indicate that kettle shape, number and design of paddles, height to diameter ratio, and speed of stirring are important variables affecting the over-all mixing efficiency of a grease kettle. A mathematical expression has been developed which permits the calculation of the mixing time of flat-dish bottom kettles having simple 2-way agitation. It is also shown that the hemispherical design of kettle 2 is the most efficient one. This kettle produces a significantly different mixing pattern, which is attributed to both the different kettle shape and the different paddle design. The contribution of the paddle design to the over-all efficiency of this kettle cannot be estimated from the available data. Its significance could be determined by incorporating certain design features found in the new hemispherical kettle into the design of the pilot plant kettle which uses the flat-dish type bottom. With the help of this radioactive tracer method, it will then be possible to determine quantitatively the contribution of the aforementioned variables to the over-all mixing efficiency of a kettle and develop a much more efficient grease kettle design.

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A. Beerbower *received his B.S. in chemical engineering from the California Institute of Technology in 1935, and his M.S. in the same field from Columbia University in 1936. After his graduation, he joined the Esso Research and Engineering company (formerly Standard Oil Development company). In 1946, he transferred to Esso Standard Oil company in Baltimore, where he is presently a petroleum technologist in charge of grease and specialities development. He is a member of the A.I.C.E., the A.C.S., A.A.A.S., and Baltimore's Applied Nucleonics Society.*

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Pyrogenic Silica-Thickened Greases

By: Peter B. Marsden
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Presented at the NLGI 25th annual meeting in Chicago, October, 1957

Abstract

THE relative merits of precipitated and pyrogenic silicas as grease thickeners are discussed. The considerable thickening effect of water as an additive is shown, followed by the results of the effect of higher boiling alcohols and diols. Other additives investigated were amines and pyrogenic alumina. Each of these additives was found to improve considerably one or two only of the three properties investigated: water resistance, shear stability, and consistency. For example: di-amines impart outstanding water resistance to pyrogenic silica-thickened greases but do not improve their shear stability or consistency.

However, one class of additives, the poly-alkylene glycols, was found to improve all three of the properties investigated. The effect of additives, in various concentrations and molecular weights, in several base oils was investigated together with the effects of premixing temperature. The findings of this laboratory study were used to make several greases on production equipment.

The results of tests on these greases by an independent testing laboratory are given, and indicate that considerable improvement in the three properties investigated can be obtained by the correct choice of additives.

Introduction

The demand for greases having higher temperature resistance than conventional soap based greases has led to considerable interest in non-soap thickeners. Silicas of very small particle size are an important class of non-soap thickeners and can be divided into two types: precipitated silica and pyrogenic silica. The former have been known and studied as grease thickeners for many years but it is only in the last few years that pyrogenic silica has been commercially available in large quantities.

This paper describes the basic differences between the two types of silica and goes on to discuss the use of pyrogenic silica in the preparation of greases. Formulations are described that overcome the earlier limitations of silica-thickened greases as far as water resistance and shear stability are concerned.

Test results on such greases indicate that pyrogenic silica is a useful addition to the family of non-soap grease thickeners.

Preparation

Precipitated silicas are prepared by the reaction of an aqueous solution of a silicate with a mineral acid. A typical case is the reaction of sodium silicate with hydrochloric acid under such conditions that a gel is not formed. The properties of the final product may be varied by controlling the temperature, concentration and rate of addition of the reactants. The important point in this method is that the silica is formed in water and must then be dried.

Pyrogenic silicas are prepared commercially by hydrolysing a silicon halide in the vapor phase. A typical example is the reaction of water vapor produced by burning hydrogen at 2000°F. with silicon tetrachloride. This method produces silica which differs in several important properties from precipitated silica. Three of these properties will be discussed:

1. Porosity
2. Nature of the Surface
3. Ease of Dispersion

I. Porosity

As can be seen from Table 1, typical pyrogenic and precipitated silicas may have the same nitrogen surface area, although the precipitated silica has a much larger particle size. This is because the precipitated particles may have an open sponge-like porous nature and the nitrogen method measures the surface area of these pores or capillaries as well as the exposed surface. Pyrogenic silica particles, however, are very much smaller and non-porous. Almost all the surface area shown is "external."

As it is the external surface area only which is involved in grease thickening, the pyrogenic silicas are usually more efficient grease thickeners than precipitated silicas.

TABLE 1
Surface Area and Porosity of Silica Thickeners

	Precipitated Silica	*Pyrogenic Silica*
Average particle size (Microns)	2.0	0.02
Calculated surface area (m^2/gm.)	1.43	143.0
Measured surface area (N_2) (m^2/gm.)	170.0	170.0
% Measured area which is external	0.8	84.0

II. Nature of the Surface

Table 2 shows that roughly 85 per cent of the nitrogen surface area of a typical precipitated silica is covered with hydroxyl (-OH) groups. These groups are

TABLE 2

Nature of the Surface of Silica Thickeners

	Precipitated Silica	*Pyrogenic Silica*
Nitrogen surface area (S_{N_2})	170.	170.
Hydroxyl surface area (S_{OH})	143.	85.
S_{OH}/S_{N_2}	0.85	0.50
Heat of immersion (in H_2O) ($Ergs/cm^2$)	340	180.

hydrophilic, or water sensitive, and are the reason for the very low water resistance of precipitated silica greases. The typical pyrogenic silica has only 50 per cent of its surface covered with hydroxyl groups and greases made with this type of silica are a little less water sensitive.

The more hydrophilic or water sensitive nature of precipitated silica is shown by its higher heat of immersion in water, which comes from the fact that there are more hydroxyl (-OH) groups to be wetted by water on immersion.

III. Ease of Dispersion

The precipitated silicas are a little more difficult to disperse in a hydrophobic medium due to the more hydrophilic nature of their surface and the fact that the particles are more strongly held together in the agglomerates. This may be due to the fact that the particles are formed in higher concentration.

Greases can be made a little more easily from pyrogenic silicas because they need less intense milling to develop their full thickening efficiency.

This paper describes the results of a program designed to improve the water resistance and shear stability of pyrogenic silica-thickened greases without sacrificing too much of their excellent thickening efficiency.

Numerous compounds were examined as additives in such greases with initial screening tests confined to resistance to boiling water and shear stabiltiy after 5,000 strokes in the grease worker. If the grease looked at all promising in either of these tests water resistance was further tested by the Navy Water Absorption Test (Mil-G-16908) and 100,000 strokes with 10 per cent added water, and shear stability by 100,000 strokes in the grease worker.

All greases made in our laboratory have been made by pre-mixing all ingredients with a propeller stirrer and then milling on a three-roll paint mill. Greases are usually given two passes through the mill—the first, a quick "open" pass and the second, at a tight setting. In both passes, the clearance between the last two rolls is set at .0015". In order to prevent condensation forming on the rolls and introducing water into the greases, hot water at 140°F. is circulated through the rolls. Most of the preliminary work was performed

EFFECT OF ADDED WATER ON GREASE CONSISTENCY

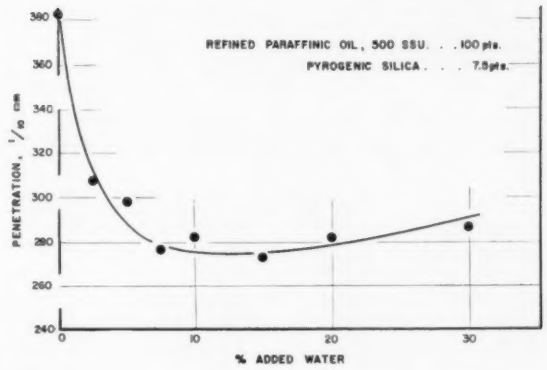

FIGURE 1

with a highly refined paraffinic oil with a viscosity of 500 ssu at 100°F. and a viscosity index of 100.

Without additives, moisture-free pyrogenic silica has certain disadvantages when used to make greases. The greases are thixotropic and have very poor water resistance. However, pyrogenic silicas normally have up to 1.5 per cent water adsorbed on their surface and this is sufficient to remove most of the thixotropy and also to improve the yield. In fact, additional water may be used to increase the yield considerably. This effect is shown in Figure 1.

The considerable thickening effect caused by the addition of water is, of course, highly desirable, but is lost when the grease is heated above 212°F. This drawback led to a study of additives which might have a similar thickening effect combined with higher boiling points. Numerous compounds were studied, particularly alcohols and diols and some useful additives were found.

EFFECT OF DUOMEEN T ON YIELD FOR No. 2 GREASE

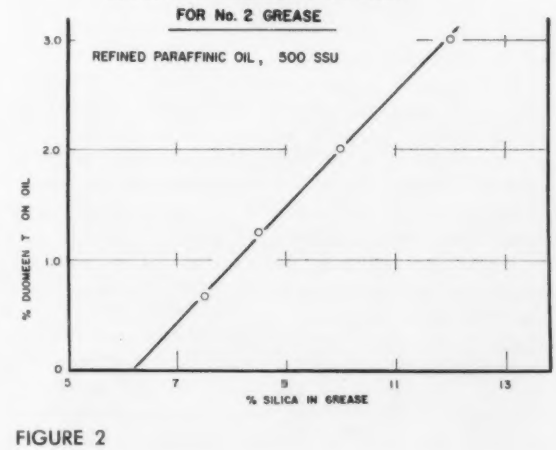

FIGURE 2

Table 3 shows six of these additives selected to illustrate the range of physical properties that may be obtained. It is interesting to note that of the three properties, thickening power, water resistance, and shear stability, two may be improved by the use of one of the additives, but none improves all three properties.

A class of additives which imparts outstanding water resistance to pyrogenic-silica-thickened greases is the fatty di-amines.

The water resistance imparted by these additives is obtained at the expense of thickening efficiency as can be seen on Figure 2. Greases can be made with pyrogenic silica and DUOMEEN T which will withstand 100 hours refluxing in boiling water with no noticeable effect. However, in this case, almost twice as much silica is needed as in an additive-free grease

TABLE 3

Effect of Certain Polyols on Grease Properties

	POLYOL					
	1	2	3	4	5	6
Penetration	377	356	336	330	310	290
H_2O resistance (minutes at 212°F)	15	9	3	0	0	0
Shear stability (% change 5,000 strokes)	2	6	7	8	9	11

7 parts Silica, 0.7 parts Polyol in 100 parts refined Paraffinic Oil–500 SSU

1. 2-ethyl–1,3-hexanediol
2. 2-methyl–1,3-hexanediol
3. Hexylene glycol
4. Trimethylol propane
5. 1,5-pentanediol
6. 1,4-butanediol

to give the same consistency. These greases also have poor shear stability.

Another additive, pyrogenic alumina, made by the same process as pyrogenic silica, is of interest because of its inorganic nature.

If a grease is thickened with a blend of pyrogenic silica and pyrogenic alumina, the water resistance and shear stability improve, as the percentage of alumina in the blend increases, but the thickening effect decreases. Thus, except for economic reasons, pyrogenic alumina by itself would be a good grease thickener. These data are shown in Figures 3, 4 and 5.

All of the additives discussed so far improve one or two of the three properties: water resistance, shear stability, and thickening power. None improves all three. Now we come to a class of additives that improves all three properties. This class compromises the polyalkylene glycols. The work in our laboratory was carried out with the particular brand known as UCON made by the Union Carbide company and

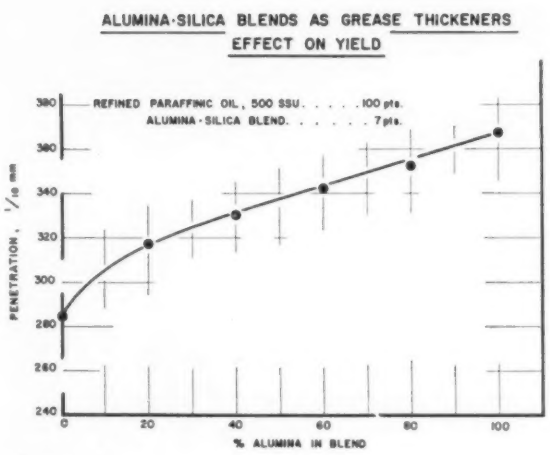

FIGURE 3

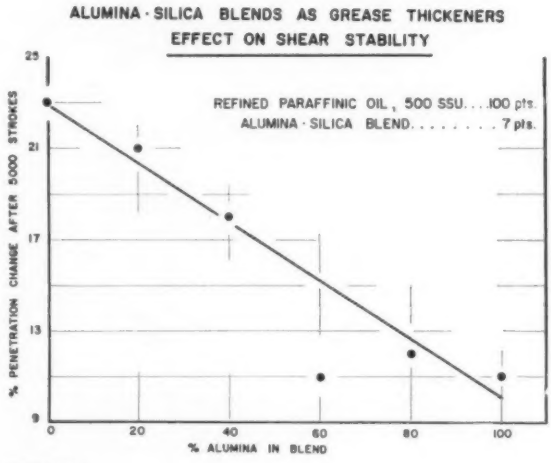

FIGURE 4

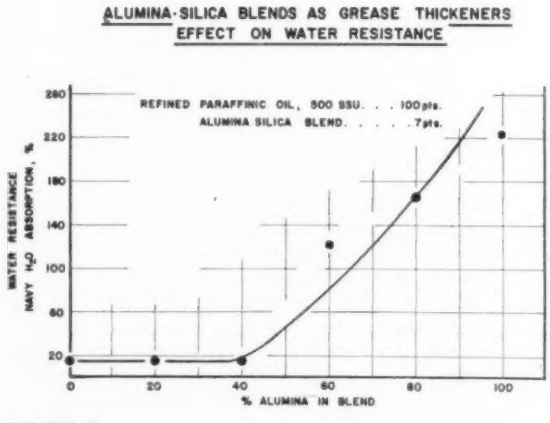

FIGURE 5

TABLE 4

Effect of Pre-Mix Temperature on Grease Properties

	Pre-mix Temperature, °F		
	250	300	350
Shear Stability (% change at 10,000 strokes)	9	6	4
Water resistance (Navy H_2O absorption, %)	30	35	40
FORMULATION			
Refined Paraffinic Oil (2000 SSU)	100 parts		
Pyrogenic Silica	8 parts		
Ucon lb 165	0.8 parts		

it will be easier if we use this designation, although it must be realized that other companies also make poly-alkylene glycols.

In order to ensure that the silica surface was moisture-free so that the Ucon could be adsorbed without interference, the pre-mixes of oil, Ucon and silica were heated, with stirring, above 212°F for a few minutes before milling on the three-roll mill. As shown in Table 4, the pre-mix temperature has an important effect. Although higher temperatures do improve shear stability and water resistance slightly, it was decided for practical reasons to use 300°F for all ensuing experiments.

The data shown on this and the following pages for Navy Water Absorption do not refer to an end-point where water is rejected as described in Mil-G-16908.

TABLE 5

Effect of Ucon Viscosity on Grease Properties

	Ucon Viscosity (SSU at 100 F)			
	165	625	1145	1715
Shear stability (% change at 10,000 strokes)	4	5	4	5
Water resistance (Navy H_2O absorption, %)	35	35	45	55
FORMULATION				
Refined Paraffinic Oil (2000 SSU)	100 parts			
Pyrogenic Silica	8 parts			
Ucon	0.8 parts			

TABLE 6

Effect of Ucon Concentration on Grease Properties

	Ucon Concentration (% on Silica)			
	5	10	15	20
Shear stability (% change at 10,000 strokes)	6	4	4	3
Water resistance (Navy H_2O absorption, %)	30	45	80	80
FORMULATION				
Refined Paraffinic Oil, 2000 SSU	100 parts			
Pyrogenic Silica	8 parts			
Ucon lb 1145	above			

TABLE 7

Effect of Oil Viscosity on Grease Properties

	Oil Viscosity, SSU		
	500	1000	2000
Shear stability (% change at 10,000 strokes)	6	5	3
Water resistance (Navy H_2O absorption, %)	80	80	80
FORMULATION			
Refined Paraffinic Oil	100 parts		
Pyrogenic Silica	8 parts		
Ucon lb 1145	1.6 parts		

In the case of Ucon-silica greases such an end-point is not reached, instead, we have taken the end-point to be where there seems to be a phase inversion to an oil-in-water emulsion.

The viscosity of the Ucon used has very little effect on shear stability, as can be seen in Table 5, but water resistance is improved by using the higher viscosity grades.

Table 6 shows the effect of varying the Ucon concentration. It can be seen that 15-20 per cent of Ucon, based on the weight of silica gives marked improvement in shear stability and water resistance.

Table 7 shows that increasing the viscosity of the base oil, when this is a refined paraffinic oil, has no effect on water resistance, but does improve shear stability.

Table 8 summarizes the effect of these four variables.

Table 9 shows the important effect of different types of oil on shear stability and water resistance of these silica-Ucon greases. The less highly refined oils give an improvement in these properties with the less highly refined naphthenic oil giving the greatest improvement.

The grease consistency obtained with varying amounts of silica is shown in Figure 6. It can be seen that only 6.5-7.5 per cent silica is needed to make a No. 1 grease and only 8-9 per cent to make a No. 2 grease.

TABLE 8

Factors Affecting Water Resistance and Shear Stability

UCON AND SILICA IN REFINED PARAFFINIC OIL

An INCREASE in:	
Pre-mix temperature results in:	IMPROVED Water Resistance IMPROVED Shear Stability
Ucon viscosity results in:	IMPROVED Water Resistance UNCHANGED Shear Stability
Ucon concentration results in:	IMPROVED Water Resistance IMPROVED Shear Stability
Base oil viscosity results in:	UNCHANGED Water Resistance IMPROVED Shear Stability

TABLE 9
Effect of Oil Type on Grease Properties

	OIL TYPE, 500 SSU at 100 °F		
	Refined Paraffinic	*Less Refined Paraffinic*	*Less Refined Naphthenic*
Shear stability (% change at 10,000 strokes	6	4	1
Water resistance (Navy H_2O absorption, %)	80	100	105

FORMULATION

Oil	100 parts
Pyrogenic Silica	8 parts
Ucon lb 1145	1.6 parts

The information obtained in this work was used to formulate a typical grease which was then made on a production mill, the Tri-Homo, and tested by an independent grease laboratory, the Phoenix Chemical Laboratory in Chicago.

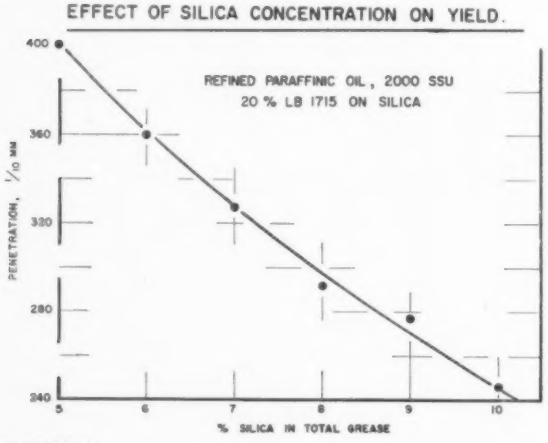

FIGURE 6

The formulation is given in Table 10 and the test results in Table 11. There are two interesting points about these results. Firstly, the oxidation stability is remarkably good considering the absence of any added oxidation inhibitor, and secondly, the excellent results of the test for deleterious particles and the Shell 4-Ball Wear Test show conclusively that well-dispersed silica greases are not only completely non-abrasive but are in fact very good lubricants.

TABLE 10
Formulation of Typical Pyrogenic Silica-Thickened Grease GR-324

Refined Paraffinic Oil (2000 SSU)	92%
Pyrogenic Silica	7%
Ucon lb 1145	1%
Pre-Mix Temperature	300 °F

Made with one pass through TRI-HOMO No. 2½ at clearance of .001"

TABLE 11
Tests Results on Typical Grease — GR 324

60 stroke penetration	326
Change at 100,000	10%
Navy H_2O absorption	80%
Water washout	3%
Leakage wheel bearing	3 gms.
Oil separation under pressure	3.9%
Dropping point	over 500°F
Oxidation stability:	
100 hours	5 psi *
300 hours	17 psi *
Deleterious particles	4 scratches
Shell roll test	63 points
Shell 4-ball wear:	
Wear spot—1200rpm—21Kgm	0.504mm
Mean specific pressure	61,000 psi

*No oxidation inhibitor added

This grease formulation is intended to show only that pyrogenic silica is of interest to the grease industry. It is not meant to be an ideal formulation.

Any specific property can be varied as shown earlier and an experienced grease formulator will undoubtedly think of many refinements.

Pyrogenic silica is an efficient, easily dispersed, grease thickener which has the advantage that by the correct choice of additives and manufacturing technique a wide range of grease properties may be produced.

About the Author

P. B. MARSDEN *is in charge of silica-thickened grease development with Godfrey L. Cabot, Inc. He took the honours degree in chemistry at the University of London and first worked for Imperial Chemical Industries on acrylic polymers and polymerization catalysts. Coming to the United States in 1952 as technical director of Landen Putty Works, he later became president of Sealube company, manufacturers of specialty greases and protective coatings. Marsden became associated with Cabot as consultant on caulking and glazing compounds and later he was appointed to the permanent staff of the pigments application group. Marsden is a member of ASTM, the Federation of Paint, Varnish and Production clubs and is active in the work of NLGI.*

ASTM Tech G News

EDITOR'S NOTE—The following material is presented as a summary of activities by ASTM's Committee D-2 on Petroleum Products and Lubricants, as a service to the lubricating grease industry. It was reviewed by Mr. C. L. Pope, chairman of Tech G, and Mr. P. R. McCarthy, secretary. It is hoped that semi-annual articles can be published in the NLGI SPOKESMAN following the ASTM June and February meetings.

A. Resume of Section IV Report on Oil Separation from Lubricating Greases in Storage

This article, in toto, is currently being examined for publication in the "ASTM Bulletin". If the publication schedule of the "Bulletin" is such that the Section IV report cannot be accepted, Technical Committee G will request that the report be released for publication in the NLGI SPOKESMAN.

A brief summary of this report was presented at the 1957 NLGI meeting.

B. Reactivation of Section II—Subsection 6

Section II of Technical Committee G has reactivated Subsection 6 on Storage Stability of Greases, with Mr. H. A. Woods, Shell Oil Company as the new Chairman.

The research of Section IV now gives the basis for standardizing a method for predicting the oil separation characteristics of greases in storage. Subsection 6 is currently engaged in cooperative test work which may eventually result in the development of such a method.

C. Mr. H. T. Peeples Appointed Second Vice-Chairman of Technical Committee G

At the June 1957 meeting Mr. H. T. Peeples, Timken Roller Bearing Company, was appointed Second Vice-Chairman of Technical Committee G.

Mr. Peeples joined Timken Roller Bearing Company in 1926 as a foreman in the laboratory. Shortly thereafter he became associated with Mr. Oscar Maag and served as his assistant until Mr. Maag's retirement in 1955. Mr. Peeples succeeded Mr. Maag as Lubrication Engineer.

In addition to his ASTM activities, Mr. Peeples is also a member of several CRC Committees and is a member of the SAE and ASLE.

D. Method of Test for Low Temperature Torque of Ball Bearing Greases

A method of test for Low Temperature Torque of Ball Bearing Greases was published as a Tentative Standard (D 1478-56 T) in the November, 1957, issue of ASTM D-2 Standards.

This method determines the extent to which a low temperature grease retards the rotation of a slow speed ball bearing when subjected to sub-zero temperatures.

Briefly the procedure is as follows: A No. 204 open ball bearing is packed completely full of the test grease. Excess grease is struck of flush with the sides of the bearing. The bearing is then attached to the shaft of a 1 r.p.m. motor assembly. The bearing remains stationary while its temperature is

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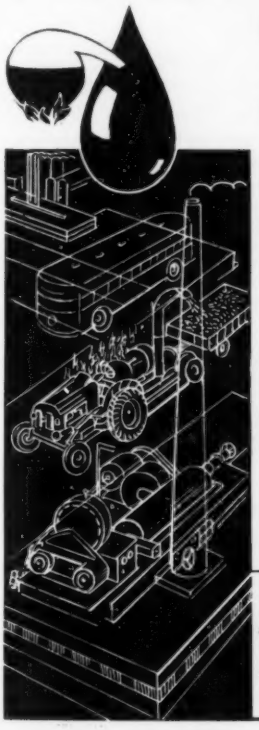

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lowered to –65° F. and held there for 2 hours. At the end of this time the inner ring of the ball bearing is rotated at 1 r.p.m. while the restraining force on the outer ring is determined.

E. Section III—Subsection 6 Organized

Subsection 6, Section III of ASTM's Technical Committee G had its organizational meeting at the February D-2 meeting in Houston, Texas. The objective of this Subsection is to consider for ASTM standardization Coordinating Research Council's L-41 Technique for evaluating rust preventive properties of lubricating greases. It was agreed at the aforementioned meeting that a round robin of tests would be undertaken shortly to investigate possible improvements in precision of the method.

F. Revision of ASTM Method D 128 "Analysis of Lubricating Grease"

The November, 1957, compilation of ASTM test methods pertaining to petroleum products incorporate revisions of interest to all who carry out analyses of lubricating greases.

New types of lubricating greases, developed in recent years, were not successfully analyzed by the procedures detailed in O-128-47. Still newer greases have been introduced recently; and advances in research leave no doubt that the field will be further expanded. It is thus unlikely that one procedure will ever encompass all the possible variations in lubricating greases.

Nevertheless, Technical Committee G, Section I, Subsection I, was assigned the task of attempting to broaden the utility of D-128-47, to include some of the newer thickeners and lubricating fluids.

Work started with a survey of procedures then being used in different laboratories. As the investigation proceeded, its ramifications increased, causing Subsection I to split into four groups. These studied procedures related to non-soap thickeners, soaps of 12-hydroxy stearic acid, ester-type fluids, and silicones. Due to diligent effort and earnest co-operation among the committee members, slow but steady progress resulted, in two directions—one a modification of D-128 which permits the analysis of soaps of hydroxy acids, the other an extraction procedure, to be used where the regular D-128 procedure is unsuitable.

The modification of D-128 consists primarily of keeping solutions warm enough (140-145° F.) to prevent precipitation of 12-hydroxy

Continued, page 238

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stearic acid from n-hexane solutions. This simple statement conceals the enormous amount of work which was required in order to specify, justify, clarify, then carry out the detailed changes.

This work brought about a further change. It was found that increasingly, analysts had been changing to n-hexane as a replacement for Grease Analysis Naptha. Solvency was comparable; boiling range, and the psychological advantage of knowing its constitution, favored n-hexane. The change in solvents did not affect analytical results. In addition, an unknown lubricating grease would best be handled with n-hexane from the start. Then if precipitation or gelation occurs, the solution need merely be warmed to 140-145° F. on the presumption that 12-hydroxy stearic acid is present. If Grease Analysis Naphtha were the solvent when such precipitation occured, replacement by n-hexane before continuing would be necessitated.

The other Section I program utilizes a different approach. An analyst attempting to analyze an unknown grease would use the procedures of D-128 or might employ preliminary tests of his own. If it is found that D-128 is not suitable for the lubricating grease at hand, then the extraction procedure should be tried. An extraction apparatus described elsewhere, for example in ASTM Method D-473, is slightly modified. The grease, in a thimble, is treated with hot n-hexane. The fluid component is extracted into the solvent layer; the thickener remains in the thimble, along with any other hexane-insoluble materials. Further steps in the analysis are left to the analyst, who is presumed to be skilled and experienced.

The extraction procedure, which appears as an Appendix I in the 1957 D-2 Manual, is being published as information only. It will permit the separation from the thickener of ester and silicone fluids; it will permit the separation from lubricating fluids of solid thickeners, or soaps of hydroxy acids. It may also be applied to certain conventional lubricating greases; but other greases cannot be so analyzed. The difficulty of delineating the bounds of this approach make D-128 the preferred procedure, the optional method being reserved to be tried on products for which D-128 has been found unsuitable.

The Proposed Optional Method will be republished for information as an Appendix to D 128, Standard Methods for analysis of grease. Comments from users of this method are invited. They may be sent directly to the ASTM, or to the chairman of the appropriate Section or Subsection. If experience proves the need for and validity of the method, it may then be incorporated as a part of D-128, equal in stature to the venerable but invaluable procedures applicable to the older formulations.

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Patents and Developments

Greases Containing N-Acyl-p-Amino Phenols As Stabilizers

Patent 2,824,838 issued to D. W. Young and D. L. Cottle, assigned to Esso Research and Engineering Company.

N-Acyl-p-aminophenols similar to those in the preceding patent are employed in amounts of 0.25-2 per cent by weight as antioxidants in lubricating greases. The grease is preferably made by adding to a heated lubricating oil a saturated fatty acid, also adding a quantity of metallic hydroxide (e.g., NaOH) sufficient to form the desired amount of grease-making metal soap of the fatty acid and also leave a portion of the acid unreacted, then adding an amount of an aminophenol sufficient to react with the unreacted acid and form the desired amount of acyl-p-aminophenol, and finally stripping the resulting mixture of water to form the stabilized grease composition. Resistance against corrosion of aluminum and magnesium alloys is claimed.

Lithium Soap-Boric Acid Ester Containing Grease

U. S. Patent 2,815,325 issued to M. J. Pohorilla and R. K. Smith, assigned to Kendall Refining Company. A lower cost, lower penetration grease containing a minimum of the more expensive lithium soap, contains 5-25 per cent lithium soap, and 5-20 per cent of a boric acid ester. A combination of 10 per cent lithium 12-hydroxy stearate with 15 per cent triphenyl or octadecyl orthoborate was found to give a good grease structure with minimum lithium soap content.

Greases Thickened with Organosilyl-Coated Siliceous Material

Patent No. 2,818,385 issued to G. B. Alexander and R. K. Iler, assigned to E. I. du Pont de Nemours and Company. Since the thickening index of a coated silica does not increase proportionately with the

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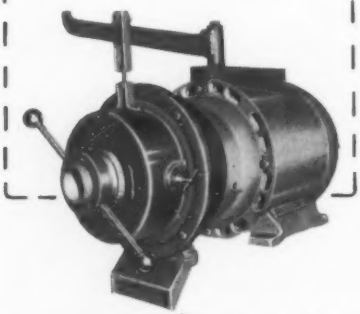

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surface coverage of the silica, there is a range where the thickening index does not change with increasing coverage of the surface until the coating amounts to about 5/6 of the complete coverage of the substrate. Thereafter, the thickening index increases as the remaining surface area is covered with organophilizing groups. A thickener consisting of a solid, siliceous substrate having a surface of silica and a surface area of from 100 to 900 square meters per gram and having chemically bonded to silicon atoms, in the silica surface, an organophilic coating consisting, at least in part, of organosilyl groups, the coating being from 1/3 to 5/6 complete, is described. The amount of coverage may be determined by the dye absorption method. The preferred coating is made up of both organosilyl and -OR groups wherein R is a hydrocarbon group of 1-8 carbon atoms in which the carbon attached to the ovygen is also attached to hydrogen, the ratio of organosilyl to -OR groups being about 1:10 to 20:1. Among the preferred organosilane compounds for coating the substrate are monomethyltrichlorosilane, phenyl chlorosilane, diphenylsilanediol, etc.

High Stability Greases

Patent 2,824,064 issued to J. M. Musselman and C. H. Whitacre, assigned to The Standard Oil company (Ohio). Greases are claimed having high consistency stability and oxidation stability, containing a mineral lubricating oil of not over 87 viscosity index, thickened by 5-50 per cent of a metal soap of a fatty acid or mixture of fatty acids preferably having 16-22 carbon atoms and 55-77 iodine value, the soap being substantially free of glycerin and glycerin reaction products and polyolefinic fatty acids, i. e., not over about 5-10 per cent.

Non-Foaming Grease-Making Process

Patent No. 2,825,692 issued to A. J. Morway and assigned to Esso Research and Engineering company. Foaming and the formation of a viscous rubbery mass have been avoided in the preparation of complex soap grease compositions by limiting the amount of lubricating oil admixed with the saponifying material during the saponification (at above 500°F.) to form a soap-oil concentrate containing 45-65 per cent soap, cooling the hot saponified product to a solidifying temperature, while maintaining it in a quiescent state and thereafter, oiling back the soap-oil concentrate to produce the finished grease of desired soap concentration.

Soap-Salt Complex Thickened Grease

Patent 2,828,262 issued to A. J. Morway and assigned to Esso Research and Engineering company. Complexes consisting of a metal salt of a low molecular weight carboxylic acid containing 1-3 carbon atoms per molecule, a metal salt of an ethylene diamine tetra aliphatic carboxylic acid containing 1-4 carbon atoms in the aliphatic acid

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group, and a metal soap of a high molecular weight carboxylic acid containing 10-30 carbon atoms per molecule are employed in producing greases suitable for a broad range of temperatures and under conditions of high unit load or boundary lubricating conditions. The low molecular weight acids may be formic, acetic, lactic, acrylic, etc. The high molecular weight acids may be the usual fatty material employed in greases, such as stearic, tallow, hydroxystearic, etc. The nitrogen-containing complex acids may be ethylene bis-imino diacetic acid sold under the name "Sequestrene" by Alrose Chemical company, or tetra- or trisodium salts sold under the name "Versene" by Bersworth Chemical company. Usually 50-97 weight per cent of lubricating oil and 3-50 per cent of soap-salt complex are employed, the latter containing 5-20 per cent of the low molecular weight acid and 1-5 per cent of an ethylene diamine tetracarboxylic acid, and 3-15 per cent of the high molecular weight acid.

Water Resistant Sodium Greases Containing Amides

Patent 2,828,263 issued to H. J. Worth and assigned to Union Oil company of California. The usual sodium soap greases can be modified by the addition of 0.5-5 per cent of an N-alkyl substituted amide of a higher molecular weight hydroxy or keto fatty acid, such as an amide product prepared by reacting under dehydrating conditions an amine selected from the class consisting of alkylene diamines having 2-8 carbon atoms per molecule and alkyl monoamines having 1-10 carbon atoms per molecule with a fatty acid such as hydroxy or keto fatty acids having 12-22 carbon atoms per molecule. A suitable material is a diamide sold under that tradename "Paricin" by Baker Castor Oil company, obtained by reacting ethylene diamine with 12-hydroxystearic acid.

Greases Containing Polyethylene Resins

Patent 2,833,718 issued to A. J. Morway, T. E. Witts and D. W. Young, assigned to Esso Research and Engineering company. Lubricating grease compositions having a high degree of structural stability and a high dropping point may be prepared with lower soap contents by incorporating into the formulations about 0.02 to 10 weight per cent of a polyethylene resin having a molecular weight of about 10,000 to 22,000 Staudinger. If, however, polyethylene resin sheets are used in the form of bags or containers to hold the metal hydroxide, the amount of polyethylene may range from 0.02 to 1.0 weight per cent. In general, as the

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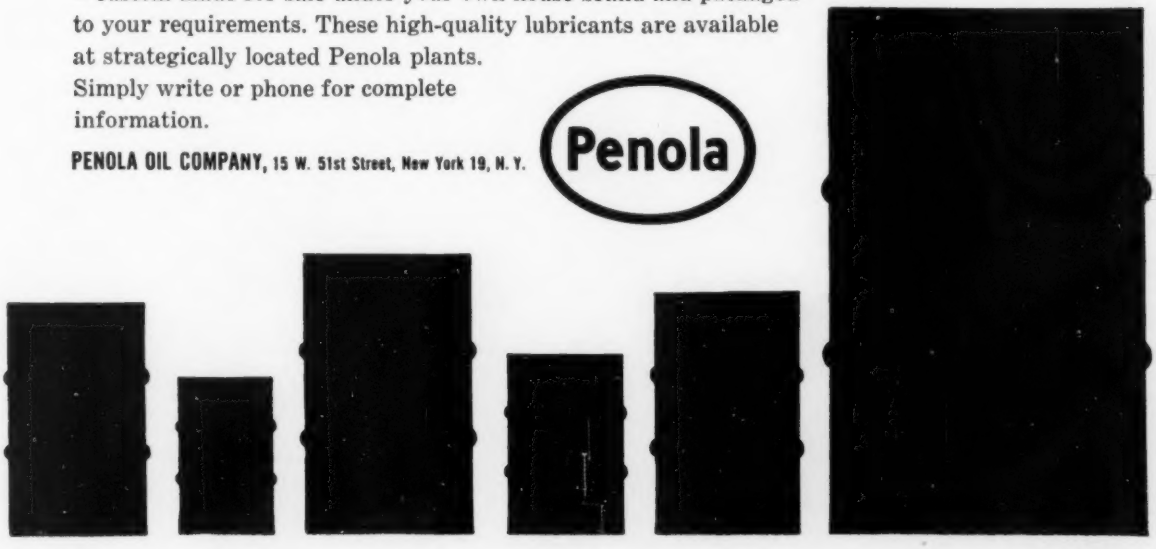


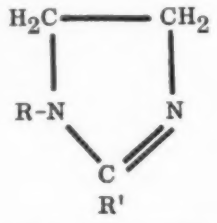
molecular weight of the resin increases, lesser amounts can be employed to obtain greases having similar structural stability.

Aerogel Greases Containing Imidazolines

U. S. Patent 2,828,260 issued to E. C. Milberger, assigned to The Standard Oil company (Ohio). Good temperature susceptibility and dynamic water stability properties are claimed of greases thickened with inorganic aerogels of silica treated with 0.25-5% of a water-soluble oil-miscible or -dispersible polyalkylene glycol diethers of 40-500 SSU at 100°F., characterized by the formula:

$$R(\text{-O-}CHR_1(CHR_2)_n\text{-})_xOR_3$$

where n is an integer having a value of preferably 1-3, x is an interger taken in sufficient number to produce an ether having the prescribed viscosity, R_1 and R_2 are hydrogen or lower alkyl groups, and R and R_3 are each lower alkyl groups. These are used in conjunction with 4-14 per cent of an alkylol or amino imidazoline which is surface-active, oil-dispersible and water-insoluble and has the following structure:



wherein R is a hydrophilic group and R' is an alkyl, hydroxyalkyl, alkylene or hydroxyalkylene group. Suitable ethers include "Ucon" lubricant, a series of polyalkylene glycols sold by Union Carbide and Carbon corporation. Suitable imidazolines include "Amine O," a commercial product identified as 1-beta-hydroxyethyl-2-heptadecenyl-imidazoline. The preferred lubricating medium is mineral lubricating oil.

Patent 2,828,261 issued to E. C. Milberger and F. Veatch is similar except that it specifies use of a polyalkylene glycol ether having one terminal hydroxy group, such as polypropylene glycol ether.

Complex of Polyalkylene Compounds with Metal Salts As Grease Thickener

Patent 2,828,265 issued to R. E. Van Strien, assigned to Standard Oil company (Indiana). By reacting polyvalent metal inorganic salts such as calcium chloride, with polyalkylene compounds, particularly the polyoxyalkylene nonionic type surface active agents, novel complexes ranging from viscous liquids to hard solids are produced, and certain of these are suitable as thickeners for greases and similar products. The salts are reacted with organic polyoxy alkylene

Continued, page 244

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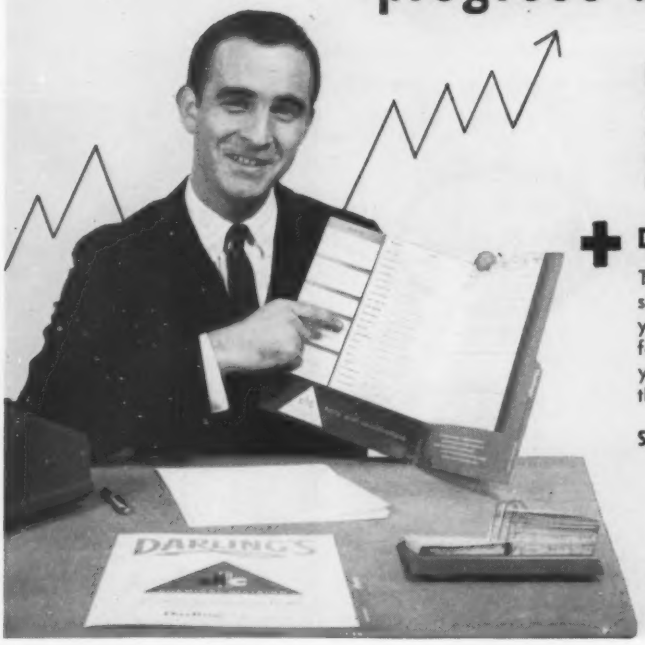

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compounds having the empirical formula:

$RX_z[(C_mH_{2m}O)_n(C_mH_{2m})X'R']_y$

wherein R may be hydrogen or an organic radical; R' may be hydrogen or an organic radical; X may be oxygen, sulfur, nitrogen,

$$-\overset{O}{\overset{\|}{C}}-O-,\ -NH-,\ -\overset{O}{\overset{\|}{C}}-NH-,$$

or $-\overset{O}{\overset{\|}{C}}-N=$

X' may be oxygen,

$$-\overset{O}{\overset{\|}{C}}-O-,\ -NH-,\ \text{or}\ -\overset{O}{\overset{\|}{C}}-NH-$$

m is 2 or 3; *n* is a positive integer from 1 to about 30; y is a positive integer from 1 to 4; and z is a positive integer from 1 to 4.

Such compounds may be produced by reacting an alkylene oxide, such as ethylene oxide, with a hydrophobic organic compound having an active hydrogen group capable of reacting with the alkylene oxide, e. g., alcohols, mercaptans, phenols, etc. The salt appears to be bound to the organic compound in a manner so as to lose its identity. For example, a complex formed from 50 parts of $t\text{-}C_{12}H_{25}S(C_2H_4O)_{11}H$ and a water solution of 30 parts of $CaCl_2$ was found to be crystalline and free of any salt or hydrate. Complex formation occurs at ambient temperatures, and the water is removed by heating the system under vacuum to about 200°F. The complex may be formed in situ in an oil fraction. An amount of 3-20 per cent by weight of complex having a ratio of salt to polyoxyalkylene compound of at least 1:4 is recommended for lubricant grease preparation. A complex resulting from the reaction of 10 grams of dioleate of a polyethylene glycol having a molecular weight of about 400, and 3 grams of $Ca(NO_3)_2$, when added in an amount of 2 per cent to a dehydrated calcium soap cup grease resulted in an increase in both mechanical stability and thermal reversibility.

NLGI SPOKESMAN

People in the Industry

Two Promotions at Foote's Research & Development Department

Recent promotions in the research & development department of Foote Mineral company have been announced by Dr. E. M. Kipp, research director, at Philadelphia.

Dr. Walter L. Borkowski has been named assistant manager of chemical research. Borkowski, who received his doctor of philosophy degree at the University of Pennsylvania, was an instructor at the school for four years. Prior to joining the Foote research staff in Berwyn, Pa., he served as supervisor of high pressure laboratory, Atlantic Refining company. His special fields of interest have been organometallic chemistry; and the preparation and properties of lithium hydride.

Mr. Douglas H. Simpson has been promoted to analytical section head. Simpson studied at McMaster university, Hamilton, Ontario, Canada, where he received a master of science degree. He also took graduate courses in analytical chemistry at Purdue university. Simpson's industrial experience includes analytical development work for the National Research council of Canada and the Mayo clinic.

Kenneth B. Wood, Jr. Appointed Assistant Manager for Climax

Appointment of Kenneth B. Wood, Jr., as assistant manager of lubricant development was announced by Reuel E. Warriner, vice president-sales of Climax Molybdenum company, a division of American Metal Climax, Inc. His responsibilities will be to develop new applications and markets for molybdenum disulphide lubricants.

Mr. Wood brings with him many years of experience in the oil industry, beginning as a trainee with the Texas company's foreign

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sales department. Early in 1951, he was recalled to active duty with the Navy for a two year period. He served with the Bureau of Aeronautics, fuels and lubricants branch in Washington, D.C., and then with the Armed Services petroleum purchasing agency also in Washington, D.C., and finally, as Navy representative to area petroleum offices in Europe and the Middle East. Upon his release from active duty he joined the Cities Service Oil company as an industrial sales engineer. For the past three years he has been manager of lubricant sales for the New York region.

Mr. Wood, a resident of Mt. Kisco, New York, is a graduate of the University of Virginia, a member of ASLE and the father of four children. He was a naval aviator during World War II and is a former lieutenant commander in the naval reserve.

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Girdler Construction Division Appoints Eastern Representative

Appointment of Kenneth D. Demarest as eastern representative of Girdler Construction division of National Cylinder Gas Company was announced by W. Roberts Wood, division president.

The division, headquartered in Louisville, Ky., designs, engineers and builds petroleum, chemical and gas processes plants. Demarest's office is in New York City.

Demarest, a native of New York City, was an engineer for Westinghouse Electric before joining Girdler Construction. He graduated from Brown university, Providence, R. I., in 1934 and attended Columbia university and Cooper Union. He is a member of the National Society of Professional Engineers and the Systems and Procedures association.

Bray Oil Co. Announces Appointment of Gerard Greco to Sales Staff

The appointment of Gerard Greco to the sales staff of the aircraft and chemical divisions of Bray Oil company was announced by Dr. Ulric B. Bray, general manager. He will serve under the direction of Robert F. Connelly, sales manager, handling the company's sodium sulfonate and government specification products.

Mr. Greco obtained his B. S. degree from the University of Alabama.

Prior to joining Bray Oil company he was in technical sales with Kelite Products Co. Inc., and Cee Bee Chemical company.

J. G. Jimenez to Replace J. R. Getty at API

J. G. Jimenez, vice president and general manager of Tidewater Oil company's eastern division, has been appointed to the general committee of the division of marketing of the American Petroleum Institute, replacing J. Ronald Getty.

William Sheffeld Appointed to Post of Treasurer of Garlock Co.

The appointment of William Sheffeld to the post of treasurer of the Garlock Packing company of Palmyra, New York, was recently announced by George L. Abbott, chairman of the board. Sheffeld is presently a vice-president.

A native of Northampton, Massachusetts, Sheffeld graduated from Dartmouth college and the Cornell Law school. He formerly served as general attorney in charge of general legal services department of the General Electric company in New York City. He succeeds George L. Abbott, who served as Garlock treasurer for twenty-eight years.

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Industry News

Emery Adds Emolein 2910 Lubricant Ester to Line

Emery Industries announces the addition of Emolein 2910 Lubricant Ester to the company's line of Emolein synthetic lubricant esters. Now fully proven in use, this new type of diester permits substantial economics in the compounding of lubricants requiring a high degree of performance over an extremely wide range of temperatures. Such lubricants include fluids for turbojet and turbo prop aircraft engines, gas turbine engine lubricants, and military and civilian all-climate greases.

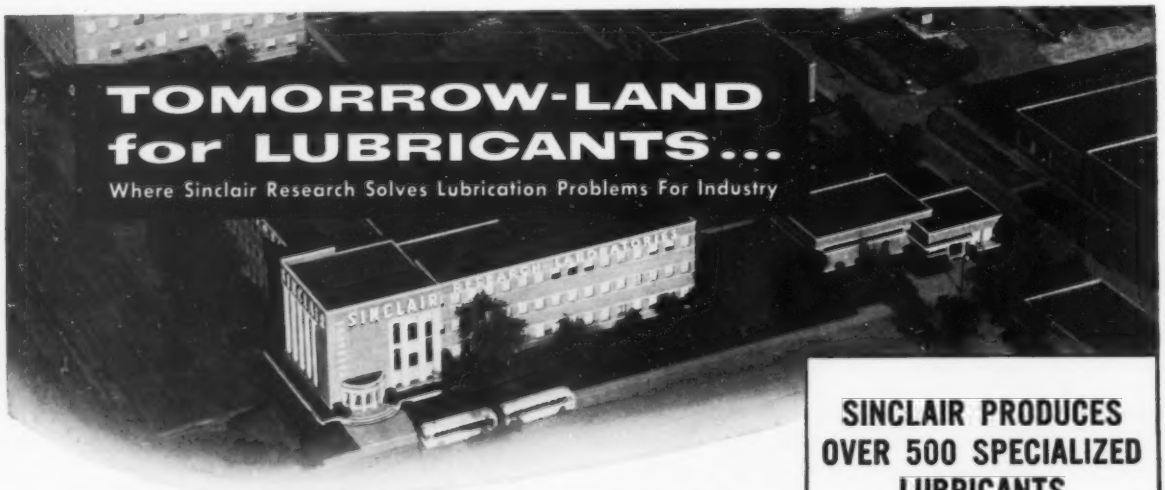
During its development period, Emolein 2910 was designated Emery 3033-S. Chemically it is diethylene glycol dipelargonate, derived from plentiful, domestic pelargonic acid. Thus, like the Emolein azelates, its supply is not dependant upon strategic imported raw materials.

Specifications and characteristics of Emolein 2910 Lubricant Ester, as well as suggested starting formulas for lubricating fluids and greases, are given in Technical Bulletin No. 411, available upon request from Emery Industries, Inc., Dept. 5, Carew Tower, Cincinnati 2, Ohio.

Nopco Moves To Newark

Nopco Chemical company and its subsidiary, Metasap Chemical company has moved its general offices to 60 Park place, Newark, New Jersey. The new headquarters will accommodate the firm's executive, accounting, advertising, and sales departments. The transfer to Newark will now provide more facilities in Harrison for Nopco's expanded laboratory activities, an essential factor in the extensive expansion program of the company. The firm's engineering, legal, production, purchasing, receiving, and shipping departments will also continue to operate at the Harrison plant.

Nopco Chemical company, which recently celebrated its 50th anniversary, produces industrial processing chemicals for a wide



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Allied Chemical Corp. to Create Separate Divisions

In the near future, Allied Chemical corporation will create two separate divisions to manufacture and market the product lines now handled by its present Barrett division, Glen B. Miller, president of Allied Chemical corporation, announced.

Barrett's long-established lines of roofing, building and paving materials will be separated from the plastics and coal chemicals and will continue to be manufactured and sold under the Barrett division name. H. Dorn Stewart will be appointed president of Barrett division. Mr. Stewart, formerly with Armstrong Cork company, joined Allied in April as assistant to the executive vice-president.

The manufacture and sale of plastics, resins and industrial chemicals will be conducted by the Plastics and Coal Chemicals division. T. J. Kinsella, head of Barrett since 1952, will be the president of that division.

This change will make possible intensified concentration on research, customer service and the development of specialized markets for each of the product lines involved. Allied's growing stake in plastics and the potential from new building materials facilities will be considerably strengthened by this realignment.

Allied is one of the leading chemical companies in the United States, operating more than 100 plants located throughout the United States and Canada and producing over 3000 products. Its principal operating divisions include Barrett, General Chemical, National Aniline, Semet-Solvay and Solvay Process, which joined together in 1920 to form Allied Chemical, and the Nitrogen division, estab-

Continued, page 250

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lished in 1952 from part of the sales organization of Barrett and certain manufacturing operations of Solvay Process. An International division was formed in 1955 to market products outside the United States.

Lincoln Adds New Air Couplers to Line of Air Accessories

Lincoln Engineering company, St. Louis, has added a new line of "Flex-O-Matic" air couplers featuring "split-second coupling action" as part of a complete line of air accessories.

The new air couplers, Lincoln engineers report, are "30 per cent lighter in weight and provide a fast, efficient means of connecting an air hose to lubricating equipment or any other air-operated devices. Also, an extra large air passage insures 35 per cent more air flow than other models."

The "Flex-O-Matic" coupler is one of many new air accessories in the "streamlined" Lincoln line, which includes standard and heavy duty lubreels, high pressure control valves, shut-off valves, lubricant pressure gauges; low pressure accessories, such as standard totalizing meters and non-totalizing meters; air line accessories, such as air blow guns, air regulators and air filters; and high pressure couplers, such as 360° hydraulic couplers.

Complete information may be obtained by writing for Catalog 65, Lincoln Engineering Co., 5702-30 Natural Bridge Ave., St. Louis 20, Missouri.

New Offices for American Potash & Chemical Corp.

Americal Potash & Chemical corporation has moved its headquarters offices into a newly constructed building at 3000 West Sixth street, Los Angeles.

Designed by Welton-Becket & Associates, the new building was constructed by Carter company on a lot adajcent to the company's previously occupied building.

The new three-story building has a total of more than 40,000 square feet of office space.

Located in the new American Potash & Chemical corporation building are all the company's executive offices including production, sales, administration, research, planning and development, defense and finance departments.

Girdler Publishes Hydrogen Booklet

Hydrogen production processes, types of plants, methods of purification, and industrial applications are described in "Hydrogen," a new booklet published by the Girdler construction division of National Cylinder Gas company, now known as the Chemetron corporation.

Continued, Page 254

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Linolenic	0.5%

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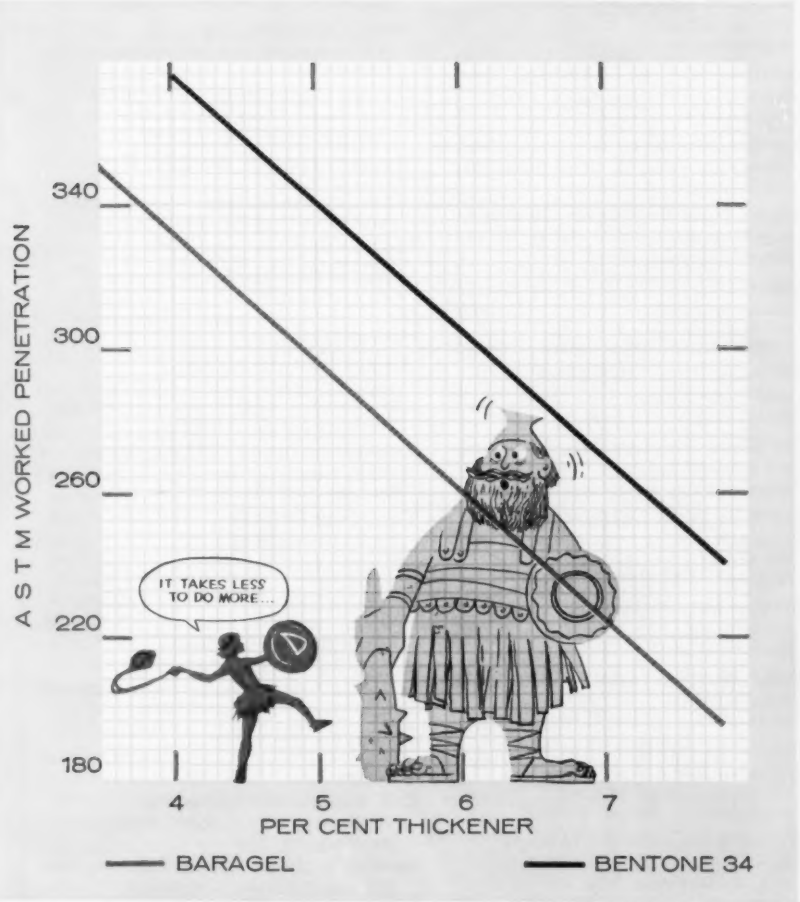
BARAGEL is considerably more EFFICIENT than Bentone 34. In many oils, 15% to 20% *less* BARAGEL is required to prepare a grease of a given consistency.



BAROID CHEMICALS, INC.

A SUBSIDIARY OF NATIONAL LEAD COMPANY

1809 SOUTH COAST LIFE BLDG. HOUSTON 2, TEXAS

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*Trademark of National Lead Co.

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Representative—Richard L. Parish, Jr.

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Representative—S. A. Bennett

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Representative—Henry Frazin

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Representative—W. J. Flint

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Representative—Neil Savee

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Representative—J. Daniel Ray

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Representative—C. K. Hubbard

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Representative—F. J. Blume

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Representative—Glenn T. Rieke

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Representative—Warren T. Trask

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Division, United States Steel Corporation
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Representative—Wm. I. Hanrahan

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P. O. Box 161, Bellwood, Illinois
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Sumner Sollitt Co.
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Representative—Lee Witzenburg

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Representative—B. A. Beaver

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Stewart-Warner Corporation
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Representative—E. G. Wicklatz

Trabon Engineering Corp.
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Representative—Hal U. Fisher

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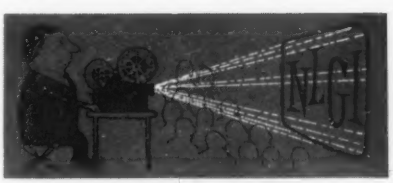

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"GREASE—THE MAGIC FILM" tells in 25 fast-moving minutes the wonderful story of lubricating grease, how this magic substance makes modern mechanical progress possible, and of the research and testing to insure a constantly better product. The 16mm movie, subsidized by NLGI, in sound and color, can also be obtained for foreign voice narration.

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Representative—Mrs. G. A. Krawetz

Products Development Laboratory
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Veresit—Fabrica de Productos Quimicos S.R.L.
Monasterio 271, Buenos Aires, Argentina
Representative—Dr. Alexander Erdely

The 36-page booklet, said by the company to be the most comprehensive treatment of the subject ever published, is a revised and updated second edition of an earlier publication by Girdler, a leader and pioneer in the development, design and construction of hydrogen plants.

(Girdler Construction Division, National Cylinder Gas Company, P. O. Box 174, Louisville 1, Ky.)

Petroleum Packaging Committee

The second meeting of the Petroleum Packaging committee of 1958 was held in Toledo, Ohio, on June 3rd. This was the beginning of the ninth year for the committee. The work of the committee for the first eight years has been very productive. Considerable time and effort has been devoted to establishing dimensional standards for containers used in the petroleum industry. Standards have been established and promulgated as listed below:

- 10 standards for metal drums and pails.
- 13 standards for shipping cases.
- 4 standards for oblong oil cans.
- 2 standards for round motor oil cans.
- 3 standards for grease cans.

The new chairman of the program subcommittee, Mr. Ed Smith, called for the chairman of all subcommittees to develop new subject matter for future meetings. He suggested the following subjects for their consideration:

Plastics as a packaging material for containers for petroleum products.

Automation in warehousing and handling of packages.

New equipment development.

Visual presentations of actual packages, slides or movies to show new developments.

A new subcommittee on traffic was created.

The name of the box car loading subcommittee was changed to shipping subcommittee. The duties of this subcommittee were enlarged to cover package and bulk shipments by all methods of transportation.

The next regular meeting will be held on October 6th, 1958, at Omaha, Nebraska. The Sheraton Fonteneld hotel will be headquarters. Socony Mobil will have charge of the arrangements.

First Brochure Tells the Story of 'Chemetron'

A new brochure describing for the first time activities of the entire company has been published by Chemetron corporation, formerly named National Cylinder Gas company.

The 32-page illustrated brochure contains sections on products and services of the company's seven divisions and its subsidiaries and

Continued, page 256

Yes — a very long way indeed! Since the first patents were granted on Axle Grease over 80 years ago, an ever increasing number of new and better lubricants has permitted equally revolutionary progress in the mechanization of industrial, automotive and agricultural equipment.

For more than a third of a century we have kept pace with these technological advances through alert research and continuous development of our production facilities.

Our complete line of WANDA Oils and Greases and our CUSTOM-MADE private label lines are quality leaders of the Oil Industry.

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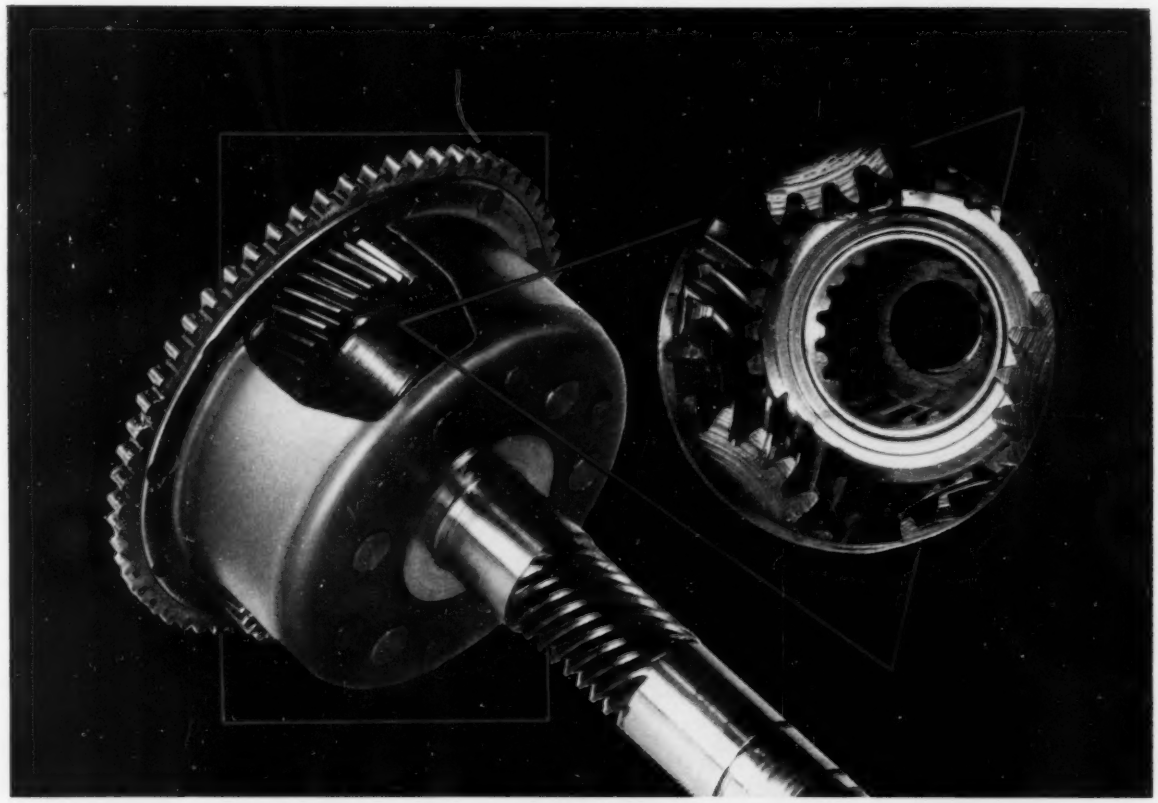

Why Nature's Miracle Additive Often Solves Toughest Lubricating Problems

Men who daily face the challenge of solving new lubricating problems or who seek better solutions to old ones frequently find the answer in ADM Spermoils. Perhaps you, too, can profit by formulating with this finest of all known natural lubricants. Here quickly, are a few of the reasons:

Sperm oil resists oxidation and is non-corrosive. Chemically, it is remarkable because it is largely made up of the higher fatty alcohol esters of the higher fatty acids. Its unsaturation is practically all mono-unsaturation. Each molecule of the ester contains but one double bond in each of the fatty acid and alcohol parts of the ester.

Two ADM sperm oils you should know more about are ADM 45° (N.W.) and ADM 38° (N.W.). N.W. is short for Natural Winter, and refers to processed oils from which solid spermaceti wax has been removed. The temperatures indicate maximum cloud points.

These two ADM Spermoils are easily sulfurized to produce non-corrosive oils containing 10-12% combined sulfur. They are miscible with all types of petroleum oils and do not separate at low temperatures. Never viscous or insoluble, they are obviously good bets for making top grade extreme pressure lubricants, crank case oils, gear lubricants, and boundary lubricants.

There's probably a way in which sperm oil can help you, too. Write Archer-Daniels-Midland, the world's largest and oldest processor and supplier of sperm oil. Ask for our 18-page bulletin 904A which gives many more technical details plus locations of technical sales people nearest you.

OTHER ADM PRODUCTS: Linseed, Soybean and Marine Oils, Paint Vehicles, Synthetic and Natural Resins, Vinyl Plasticizers, Fatty Acids and Alcohols, Hydrogenated Glycerides, Foundry Binders, Industrial Cereals, Vegetable Proteins, Wheat Flour, Dehydrated Alfalfa, Livestock, Poultry Feeds, Olefins and Hydrocarbons.

Archer-Daniels-Midland

CHEMICAL PRODUCTS DIVISION
741 Investors Building
Minneapolis 2, Minnesota

Chemifats from Nature's Wondrous Warehouse

Continued from page 254

affiliate companies and features the first history of the company.

In a foreword Charles J. Haines, president of Chemetron, explains why the company decided to change its name and realign its organizational structure, as described in the brochure.

Copies may be obtained without charge from Chemetron corporation, 840 North Michigan avenue, Chicago 11, Ill.

A-D-M's New Catalog Now Available

The chemical products division of Archer-Daniels-Midland company announces publication of a new 52-page catalog for its line of aliphatic chemicals.

Included in the booklet are test data on ADM's olefins, hydrocarbons, fatty alcohols, hydrogenated and vegetable fatty acids, glycerides and sperm oil products.

The catalog also contains the first commercial presentation of gas chromatographic composition data on olefins, hydrocarbons, fatty alcohols and hydrogenated fatty acids. Solubility, compatability and physical constants charts are given for the various products.

Intended as a technical reference for the experienced user of chemifats—fatty chemicals derived from vegetable, animal, fish and sperm whale fats and oils—the booklet also is an introduction to the chemistry involved in working with these versatile chemicals for those just beginning to use them.

Requests for copies of the new ADM catalog should be addressed on company letterhead to the chemical products division, Archer-Daniels-Midland company, Box 839, Minneapolis 40, Minnesota.

Lincoln Engineering Markets New Pressure Primer and New Material Dispensing Pump

Lincoln Engineering company, St. Louis, is offering a new air-operated pressure primer, the 'Dyna-Ram," and a new air-operated forced induction pump, the "Pile Driver," as part of their new line of industrial lubrication equipment.

"Dyna-Ram," Lincoln officials report, includes "exclusive design and engineering features that provide unexcelled performance and economy. In addition to the usual fluid materials these pumps handle extra heavy lubricants and "non-flowing compounds.' It completely empties and cleans 55 gal. drums, even dented ones, right to the bottom."

The "Dyna-Ram" is especially suited for use with the new line of Lincoln "Power-Master" air-motor operated drum pumps. Special features listed by the company include extreme ease of operation, maximum power, rugged steel construction, and jam-proof operation.

The "Pile Driver" is a maximum power unit designed to pump even the heaviest materials, reports Lincoln.

Complete information on the new Lincoln line may be obtained by writing for Catalog 65, Lincoln Engineering company, 5702-30 Natural Bridge avenue, St. Louis 20, Missouri.

CONSTANT SEARCH for BETTER LUBRICANTS by DENCO

ASSURES QUALITY IN YOUR PACKAGE

The same "Know-How" that makes Denco the first in the manufacture of bentone lubricants, is used in everyday production of Denco's complete line of greases, oils, and compounds for every lubricating and metal-working function. A "Know-How" that's backed by fifty years of experience.

We will produce to your specifications, package, label, and ship according to your instructions.

Call OL. 1-6600 or write today

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LET US MODERNIZE YOUR PLANT

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"In Engineering it's the People that count"

HARSHAW LEAD BASE

Harshaw Lead Base, as an additive to petroleum lubricants, improves extreme pressure characteristics and imparts the following desirable properties:

- Increased film strength
- Increased lubricity
- Improved wetting of metal surfaces
- A strong bond between lubricant and metal surfaces
- Resistance to welding of metals at high temperatures
- Moisture resistance and inhibits corrosion

Harshaw Lead Bases are offered in three concentrations to suit your particular needs:

Liquid	Liquid	Solid
30% Pb	33% Pb	36% Pb

Other metallic soaps made to your specifications. Our Technical Staffs are available to help you adapt these products to your specific needs.

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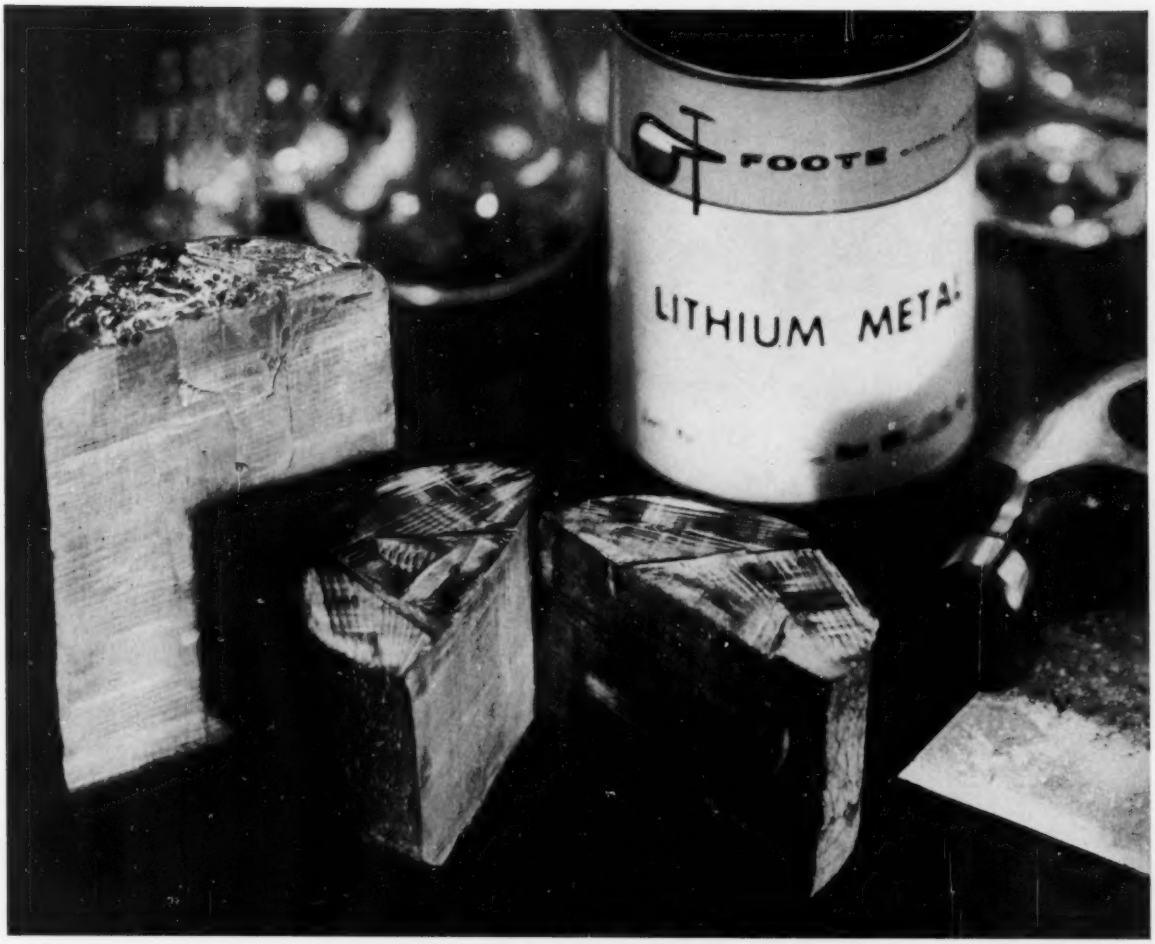

Lithium is the alkali metal with the most-*est*

Atomic weight, light*est* . . . ionic radius, small*est* . . . specific gravity, low*est* . . . bonding energy, strong*est* . . . heat capacity, high*est* . . . heat of vaporization, great*est* . . . reaction with water, slow*est* . . . reaction with hydrogen, fas*test* . . . liquid range, wid*est*.

That's a good bit of the lithium metal story right there! But it's not all. The part that should interest you most is the many present and future uses of lithium's *est's* in the chemical industry.

As the alkali metal with the widest liquid range, lithium shows great promise as a super heat transfer agent. Lithium also has the necessary high heat capacity and good thermal conductivity.

In the polymerization of olefins, lithium metal is showing unique catalytic properties distinct from that of the other alkali metals, especially in the polymerization of isoprene to natural-type rubber.

Organolithium compounds made from lithium metal are used as alkylating agents in Grignard-type reactions.

Lithium metal is also used to prepare lithium hydride which in turn is used in synthesizing simple and complex hydrides. Presumably, it is the very slight solubility of the lithium hydride above that of the other alkali hydrides that makes lithium hydride so useful.

It stands to reason . . . if you're looking for the most-*est* in an alkali metal, you should investigate lithium . . . you should read our Bulletin 101 *Lithium Metal*. For your copy, write Technical Literature Dept., Foote Mineral Company, 402 Eighteen West Chelten Building, Philadelphia 44, Pennsylvania.

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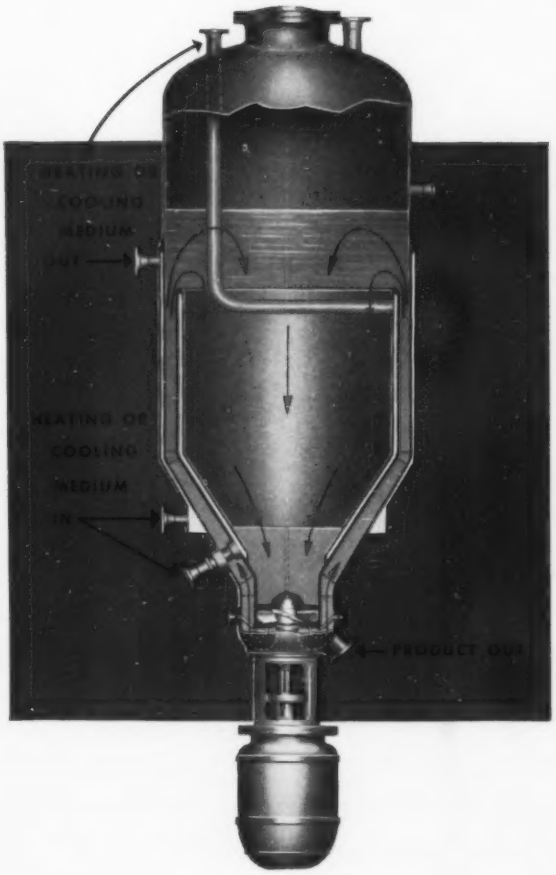

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